Chemical Engineering Journal 157 (2010) 304–310

Contents lists available at ScienceDirect

Chemical Engineering Journal

journal homepage: www.elsevier.com/locate/cej

The influence of support layer structure on mass transfer in pervaporation of composite PDMS–PSF membranes

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ARTICLE INFO

Article history:
Received 5 May 2009
Received in revised form 16 October 2009
Accepted 29 October 2009

Keywords:
Pervaporation
Composite membrane
Support layer structure
Mass-transfer resistance
Average parallel flow model

ABSTRACT

In this study, a series of composite polydimethylsiloxane (PDMS)–polysulfone (PSF) membranes were prepared to investigate the influence of support layer structure on the mass transfer in pervaporation (PV). By varying polymer concentration in the casting solution and the evaporation time in the air, phase-inversion PSF membranes with different structures and resistances were obtained as the support layers. The results indicated that the support layer could dominate the PV performance of the composite membrane when it showed a significant resistance. An average parallel flow model was proposed to evaluate the influence of support layer structure on mass-transfer resistance in composite membranes. The inhomogeneous structure of the support layer could produce extra mass-transfer resistance when the resistance of the support layer could not be negligible.

1. Introduction

Pervaporation is a separation process based on selective transport inside a dense membrane associated with vaporization of the permeant. It has been widely recognized in wastewater treatment and food industry [1–3], such as aroma compounds recovery or concentration in fruit juices [4,5] and dealcoholization of wine or beer [6,7].

In general, from a manufacturing point of view, most of the membranes applied in pervaporation are composite membranes, which consist of a very thin, selective, dense top layer, i.e. the active layer, and a porous support layer. Ideally, the active layer should provide the excellent flux and selectivity, while the support layer should exhibit the outstanding mechanical stability and not interfere with the mass transport. However, several studies have clearly demonstrated that the support layer can have significant effects on pervaporation [8–17]. Scholz [9] observed that both flux and separation factor changed when he used zeolite-filled silicone membranes with different support materials to remove ethanol from water. Similarly, in the study by Feng and Huang [10], it was concluded that the relatively tight porous support produced from a water-selective plastic could change the separation factor of the composite membrane. When Vankelecom [17] studied the effect of intrusion of PDMS in Zirfon® and PVDF asymmetric support layers, he observed that the normalized fluxes of the composite membrane changed while the selectivity remained when different pre-treatment was applied for the support layer.

In addition, there have been some models proposed to describe the influence of the support layer. One of the most accepted models was proposed by Henis and Tripodi [18] in 1980 for gas transport through composite membrane. The Henis and Tripodi model divided the flux through the interface layer, which connected the top layer and the porous support layer [17,19], into a flux through the support layer material and a flux through the selective polymer filled pores. The overall resistance could be expressed as Eq. (1):

$$R_m = R_a + \frac{R_{s1}R_{s2}}{R_{s1}+R_{s2}} \tag{1}$$

where R_a is the average resistance of the active layer, R_{s1} and R_{s2} are the resistances to the gas flow through the polymer matrix and the pore. This model was adopted in the study of Feng [10] and O. Trifunovic [20]. Fouda et al. [21] extended the Henis' model and proposed Wheatstone-bridge model for gas separation. The parallel flow model, where the resistance of the cross flow in Wheatstone-bridge configuration is equal to infinity, could explain well the experimental data. The overall resistance in parallel flow model could be expressed as Eq. (2):

$$R_m = \frac{(R_{a1}+R_{s1})\times(R_{a2}+R_{s2})}{R_{a1}+R_{s1}+R_{a2}+R_{s2}} \tag{2}$$

where R_{a1} is the resistance for the flow of the gas through the active layer that covers the area occupied by the polymer matrix of the support layer, R_{a2} is the resistance for the flow of the gas through the

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doi:10.1016/j.cej.2009.10.060

Nomenclature

R_{ai}	the resistance of top layer in the unit i (m^2 h/g)
R_{si}	the resistance of support layer in the unit i (m^2 h/g)
$C_{i,f}$	weight fraction of component i in the feed (g/g)
$C_{i,p}$	weight fraction of component i in the permeate (g/g)
J_i	the permeate flux of component i (g/m^2 h)
$R_{bl,f}$	the resistance in boundary layer in feed side (m^2 h/g)
$R_{bl,p}$	the resistance in boundary layer in permeate side (m^2 h/g)
R_m	the resistance in membrane (m^2 h/g)
R_{tot}	the total mass-transfer resistance through the membrane (m^2 h/g)
R_{sum}	the sum of average resistance of the top and support layer (m^2 h/g)
$\overline{R_{\text{sup}}}$	the average resistance of the support layer (m^2 h/g)
$\overline{R_{top}}$	the average resistance of the top layer (m^2 h/g)
x_i	the molar fraction of component i in the feed (mol/mol)
y_i	the molar fraction of component i in the permeate (mol/mol)

Greek letters

α	separation factor

active layer that covers the area occupied by the pores of the support layer. This model was also applied in the study of Gudernatsch [22] and Lipnizki [19].

The majority of previous studies [18–22] on the influence of support layer in pervaporation were performed using support layers with small resistances, which exists mostly in the interface layer. Little information concerned the influence of support layer with significant resistance. In this paper, a series of composite PDMS–PSF membranes were prepared with quite different structures and considerable resistances in the support layer. An average parallel flow model was established and used to analyze experimental data.

2. Theory

Generally, based on solution-diffusion mechanism, the flux through a membrane can be described by:

$$J_i = \frac{1}{R_{tot}}(C_{i,f} - C_{i,p}) \tag{3}$$

where $C_{i,f}$ and $C_{i,p}$ is respectively the concentration of component i in feed and permeate bulk of the membrane, and R_{tot} is the total mass-transfer resistance through the membrane. According to the resistance-in-series model, the total resistance can be expressed as:

$$R_{tot} = R_{bl,f} + R_m + R_{bl,p} \tag{4}$$

where $R_{bl,f}$, R_m and $R_{bl,p}$ is the resistance in boundary layer in feed side, membrane and boundary layer in permeate side.

It has been proved in our previous work that as for the same membrane module, R_{tot} is very close to R_m when the feed flow is over 120 L/h with a Reynolds number of 1044 [23,24]. Considering that experiments are often operated under very low downstream pressure, the concentration of component i in permeate bulk of the membrane could be neglected. Then Eq. (3) can be simplified as:

$$J_i = \frac{C_{i,f}}{R_m} \tag{5}$$

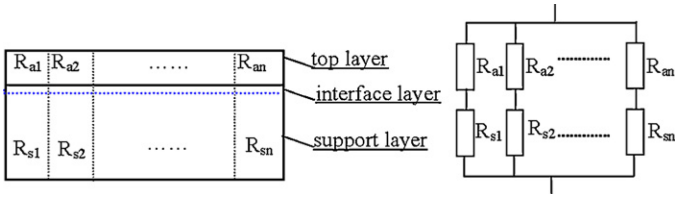

Fig. 1. Resistance unit in composite membrane.

The separation factor of component i through the composite membrane is expressed as:

$$\alpha = \frac{y_i/y_j}{x_i/x_j} \tag{6}$$

where x and y is the molar fraction of the component in the feed and permeate side respectively.

In this paper, an average parallel flow model is proposed and schematically illustrated in Fig. 1. The composite membrane is divided averagely into several elementary units that have completely the same shape. If the resistance of top layer and support layer in unit i is defined as R_{ai} and R_{si}, the overall mass-transfer resistance in the composite membrane can be described by:

$$\frac{1}{R_m} = \sum_{i=1}^{n} \frac{1}{R_{ai} + R_{si}} \tag{7}$$

The average resistance of the top layer and the support layer can be expressed as:

$$\frac{1}{\overline{R_{top}}} = \sum_{i=1}^{n} \frac{1}{R_{ai}} \tag{8}$$

$$\frac{1}{\overline{R_{\text{sup}}}} = \sum_{i=1}^{n} \frac{1}{R_{si}} \tag{9}$$

If and only if $R_{a1} = R_{a2} = \ldots = R_{ai} = R_a$
and $R_{s1} = R_{s2} = \ldots = R_{si} = R_s$

we can get

$$R_m = \overline{R_{top}} + \overline{R_{\text{sup}}} \tag{10}$$

Otherwise, the overall resistance of the composite membrane R_m cannot be expressed as the sum of the average resistance of top layer and support layer, which is noted as R_{sum} in this paper. The discrepancy between the real resistance of the membrane R_m and the sum of the average resistance R_{sum} mainly results from the difference of the resistance in structural units of the composite membrane. Therefore, the degree of the discrepancy can characterize effectively the influence of inhomogeneous structure of the membrane on the mass transport.

To simplify the calculation, the composite membrane is assumed to be divided averagely into two structural units. According to Eq. (9), the average resistance of the support layer can be written as:

$$\overline{R_{\text{sup}}} = \frac{R_{s1}R_{s2}}{R_{s1} + R_{s2}} \tag{11}$$

Due to the uniform structure of the dense top layer, the resistance of top layer can be assumed as: $R_{a1} = R_{a2} = R_a$

$$\overline{R_{top}} = \frac{R_a}{2} \tag{12}$$

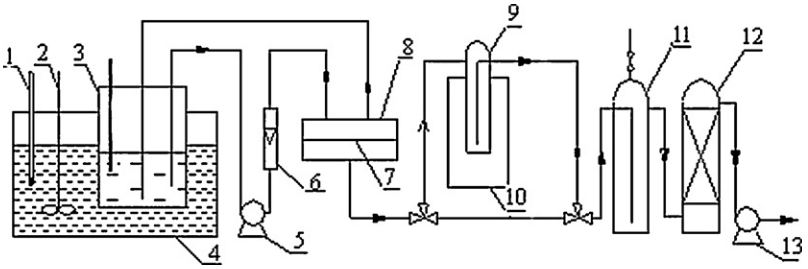

Fig. 2. PV experimental setup. (1) Thermometer; (2) puddler; (3) reservoir; (4) thermostat; (5) pump; (6) flowmeter; (7) membrane; (8) membrane cell; (9) collecting bottles; (10) refrigerators; (11) safe bottle; (12) drying bottle; (13) vacuum pump.

Further, the real resistance of the membrane R_m and the sum of the average resistance R_{sum} can be written as:

$$R_{sum} = \overline{R_{\text{sup}}} + \overline{R_{top}} = \frac{R_{s1}R_{s2}}{R_{s1}+R_{s2}} + \frac{R_a}{2} \tag{13}$$

$$R_m = \frac{(R_a+R_{s1}) \times (R_a+R_{s2})}{(R_a+R_{s1})+(R_a+R_{s2})} \tag{14}$$

Eq. (13) is very similar to Eq. (1) that represents the Henis and Tripodi model. Then, the difference between R_m and R_{sum} can be deduced as:

$$\Delta R = R_m - R_{sum} = \frac{R_a(R_{s1}-R_{s2})^2}{2(R_{s1}+R_{s2}+2R_a)(R_{s1}+R_{s2})} \tag{15}$$

It can be concluded from Eq. (15) that when the support layer has a very homogeneous structure, the resistance of each unit should be equal, and therefore the resistance of the composite membrane will be equal to the sum of the average resistance of top layer and support layer. Otherwise, R_m should exceed R_{sum}. And, the more differences exist in the structure of membrane units, the more value of ΔR will be. It is obvious that the Henis and Tripodi model could be regarded as the special case of the average parallel flow model. Consequently, ΔR could be used to evaluate the influence of the support layer, especially its structure, on the mass-transfer resistance in the composite membrane. It implies that the inhomogeneous structure of the support layer could produce extra mass-transfer resistance when the resistance of the support layer could not be neglected. Therefore, as for a certain composite membrane, it is beneficial to choose a proper support layer to reduce the extra resistance caused by inhomogeneous structure.

3. Experimental

3.1. Pervaporation membranes

In this study, the PSF membranes prepared by a phase inversion process were used as the support layer in composite PDMS–PSF membranes. The PSF concentrations in the casting solutions and evaporation time of the casting films in the air were changed to produce different PSF porous membranes with different kinds of structures. The concentration of PSF in the casting solution was varied from 12 to 20 wt.% with N-methyl-2-ketopyrrolidine (NMP) as the solvent. After the mixture was spread uniformly on a glass plate, the casting film was evaporated in the air for a certain time (0–30 min), and then immersed into a container filled with water and lasted 1 h at room temperature. Finally, being taken out of the container, the films were dried in the air for about 24 h and then were further dried in a vacuum oven for 4 h at 70 °C and 6.7 kPa.

The top layer of all the composite membranes in this study was PDMS with average molecular weight of 70,000–80,000 supplied by Shanghai Synthetic Resin Company, China. The thickness of the PDMS layer was around 6 μm for all of the membranes. The PSF with average molecular weight of 50,000–60,000 was purchased from Shanghai Shupeng Engineering Plastic Company, China. The preparation method of the composite PDMS membrane was described in our previous work [24] in detail.

3.2. Experimental procedure

Binary aqueous solution with ethanol concentration of 5 wt.% was prepared as the feed solution for all the experiments. PV experiments were performed on a continuous apparatus shown in Fig. 2. In all the experiments, the feed temperature was maintained at 40 °C and the downstream pressure was at 260 Pa. In addition, the feed flowrate was kept at 300 L/h and the effective area of the membranes is 0.00636 m^2. The system was equilibrated for 3 h before the measurements were made. In order to ensure the reliability of experimental data, each experiment was repeated three times until the data of both flux and separation factor were within an error of 5%.

3.3. Analytical methods

A densimeter (DMA5000, Anton Paar, Austria) was used to measure the liquid densities (the accuracy of 0.000001 g/mL) and to determine the ethanol concentration (the accuracy of 0.001 wt.%). Scanning electron microscopy (SEM, Hitachi S-800) was used to observe the structure of the support layer and to measure the thickness of the top layer.

4. Results

4.1. The change of PSF concentration in the casting solution

As we all know that the polymer concentration in the casting solution has great effects on the porosity of the asymmetric support membrane [25]. In general, the higher the polymer concentration is, the lower the porosity would be. In this study, the PSF concentration was changed from 12 to 20 wt.%, and the evaporation time was approximately 0 min. The PV performance of the uncoated PSF support membranes was shown in Table 1. When the PSF concentration was 12 and 13 wt.%, there was a very large amount of permeate and it was difficult to measure the flux and the separation factor exactly. That means a negligible mass-transfer resistance in PSF membranes of 12 and 13 wt.%. With the increasing concentration of PSF in the casting solution, both the flux and separation factor decreased. When the PSF concentration reached 20 wt.%, the sepa-

Table 1
PV performance of the uncoated PSF support membranes.

PSF wt.%	12	13	15	17	20
J/[g/(m^2 h)]	–	–	24280	4822	2186
α	–	–	1.27	1.24	0.85

Note: "–"means that the permeate fluxes were too much to measure.

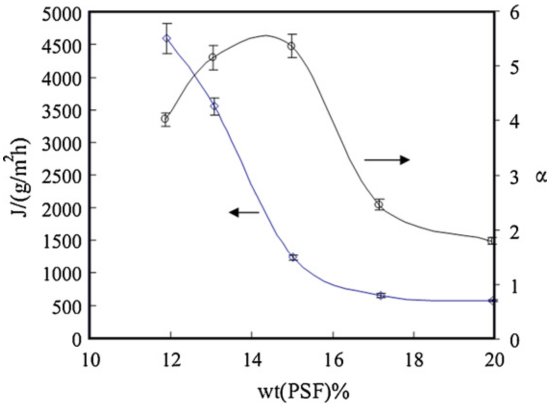

Fig. 3. Effect of PSF concentrations on the PV performance of PDMS–PSF membranes.

ration factor was merely 0.85 and the support layer was obviously selective to water.

The composite PDMS membranes were prepared using these PSF membranes as the support layer in the same condition. The thickness of the active layer was around 6 μm for all of the membranes. Fig. 3 showed the PV performance of these composite membranes with different PSF concentrations. As can be seen from Fig. 3, when the PSF concentration increased from 12 to 20 wt.%, the flux of the composite membranes decreased significantly from 4580 g/(m^2 h) to 569 g/(m^2 h). In contrast, the separation factor showed a maximum value of 5.32 with the PSF concentration between 13 and 15 wt.%. As for PSF concentrations of 20 wt.%, the separation factor was only 1.79. Therefore, it was obvious that the excessive polymer concentration of the support layer in casting solution could make the support dominate the performance of the composite membrane.

The mass-transfer resistance in the composite membranes was shown in Fig. 4. As for the uncoated support membranes with PSF 12 and 13 wt.%, the resistances were assumed as zero since the permeate fluxes were too much to measure. Therefore, the resistance of the top layer was determined as the average in the composite membranes of PSF 12 and 13 wt.%. It can be found in Fig. 4 that when the PSF concentration increased from 15 to 20 wt.%, the resistance in the supports increased rapidly and exceeded far away from the resistance of top layer. Moreover, the total resistance of the composite membranes was much more than the sum of average resistance of the top and support layer. According to Eq. (15), the great differences between R_m and R_{sum} implied that the structure of the support layer was seriously inhomogeneous and consequently resulted in an extra increase of the total resistance in composite membranes.

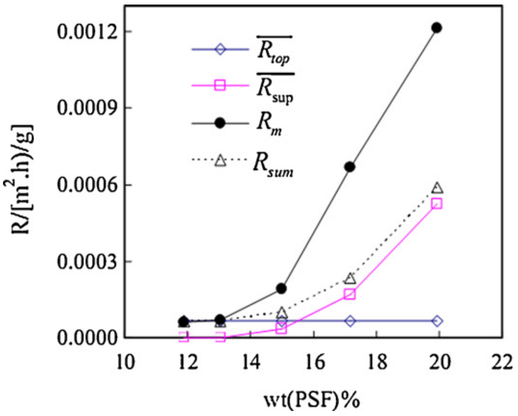

Fig. 4. Effect of PSF concentrations on mass-transfer resistance in composite PDMS–PSF membranes

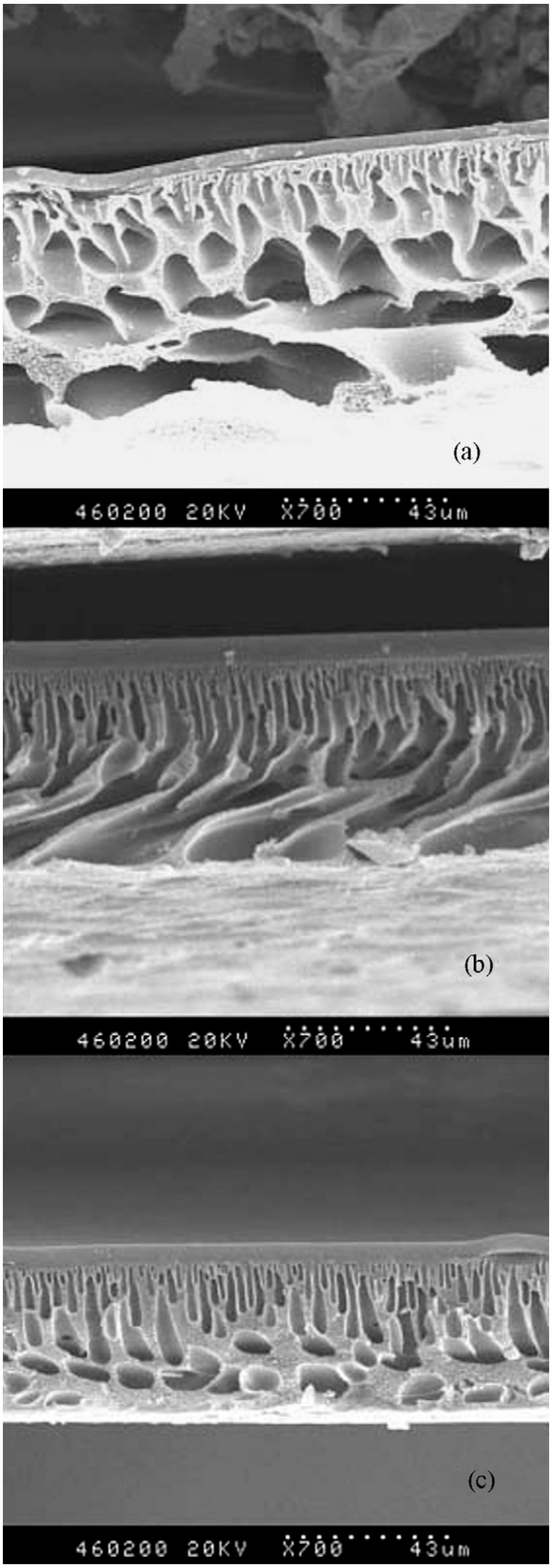

Fig. 5. Cross-sectional photograph of PDMS–PSF membranes by SEM. The PSF concentration in the casting solution: (a) 12 wt.%, (b) 13 wt.%, (c) 20 wt.%.

The SEM graphs could demonstrate very well the morphology of the support layers. As can be seen from Fig. 5, when the PSF concentration in the casting solution was only 12 wt.%, as was shown in graph (a), the pores in the membrane were wide and short, and

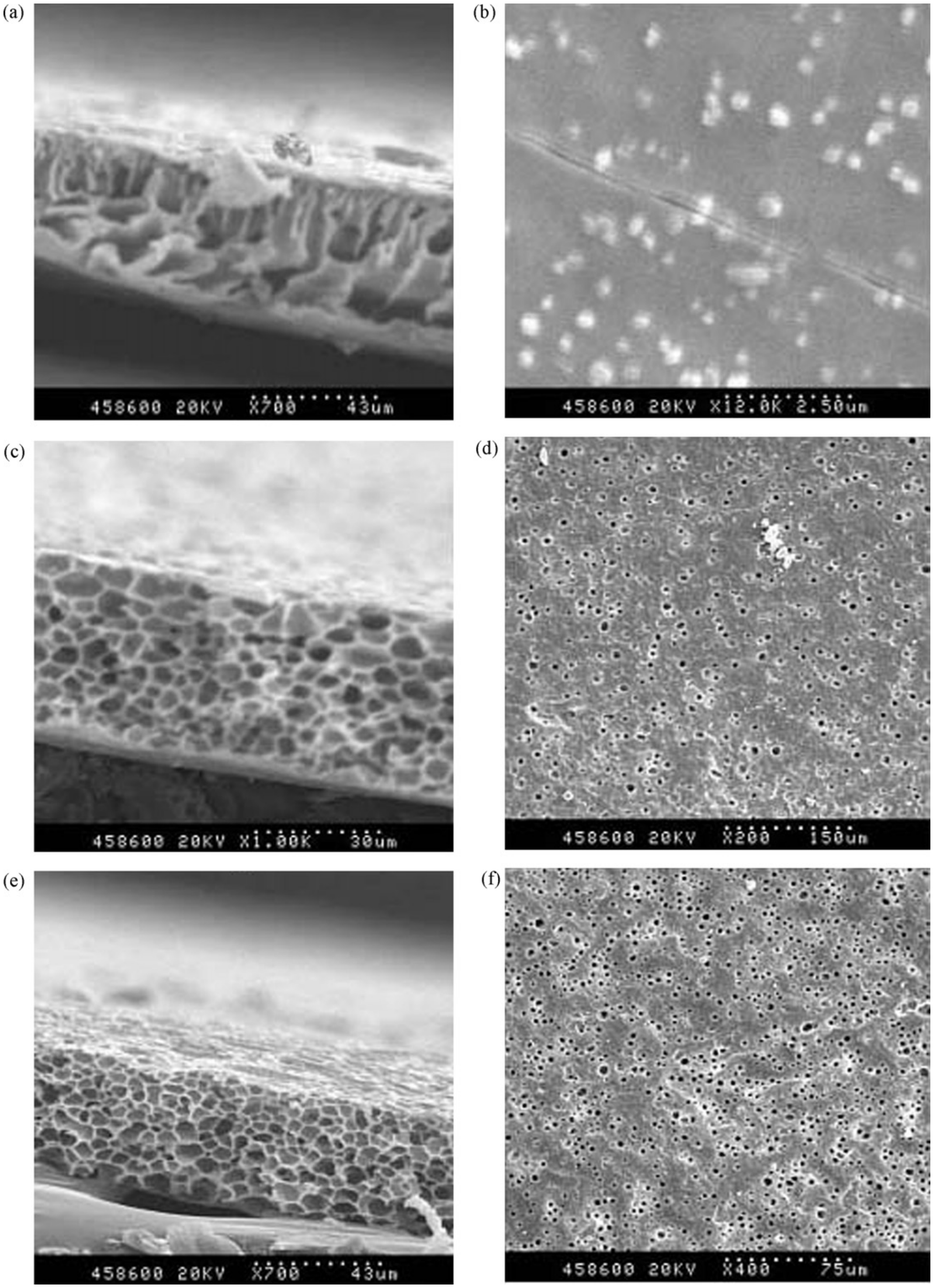

Fig. 6. SEM micrographs of 13 wt.% PSF support membrane under different evaporation time. Cross-sectional view: (a) 1 min, (c) 10 min, (e) 30 min. Surface view: (b) 1 min, (d) 10 min, (f) 30 min.

the bottom of the membrane was collapsed with very large pores. It showed a relatively high porosity contributing to the support layer a negligible resistance. However, the casting solution exhibited a very low viscosity that caused the support layer inferior mechanical intensity and many defects. Thereby, the very high flux and low separation factor of the composite membrane with PSF concentration 12 wt.% might be attributed to these flaws. When the PSF concentration was 13 wt.% in graph (b), there produced large finger typed pores from the top of the membrane directly through the bottom. The casting solution was in a quite proper viscosity and few defects produced in support layer. Graph (c) showed the structure of asymmetric membrane with PSF concentration of 20 wt.%. There were two kinds of pores in the support. In the top side, the pores are finger typed with small size; while at the bottom, there exist some large round holes. This kind of structure caused many dense parts and increased the resistance of the support layer consequently. Moreover, the very inhomogeneous structure of the support layer together with the high resistance further caused the correspond-

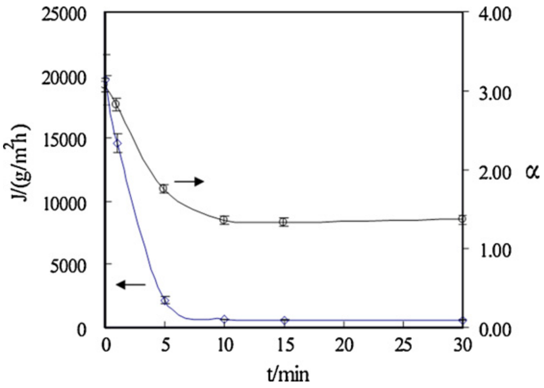

Fig. 7. Effect of evaporation time on PV performance of PSF support membranes.

ing composite membrane a rather higher resistance than the sum of the average of the top and support layer.

4.2. The change of evaporation time in the air

The evaporation time of solvent in the air is another important condition for the structure of the PSF membrane. In this study, a batch of support membranes was produced with PSF concentration of 13 wt.% under different evaporation time from 0 to 30 min. The SEM graphs were shown in Fig. 6. When the film was evaporated in the air for only 1 min, there fabricated finger typed pores and a very thin dense skin on the top of the membrane with the thickness of 0.3–1 μm. However, when the evaporation time was 10 and 30 min, the pores in the membranes changed to be spongy and showed different size. As can be seen from SEM graphs in Fig. 6, the average pore diameter with evaporation time of 10 min was bigger than 30 min evaporation, while the surface porosity was obviously less than the latter.

Pervaporation experiments were carried out with the uncoated PSF support membranes and the respective composite PDMS–PSF membranes. As can be seen from Figs. 7 and 8, with evaporation time increasing from 0 to 10 min, both the flux and separation factor decreased rapidly in PSF supports and the composite PDMS/PSF membranes. When the evaporation in the air was more than 10 min, the PV performance changed little with time, both for uncoated PSF supports and composite PDMS–PSF membranes. The fluxes were quite small and the separation factor for ethanol was only about 1–2.5. It was clear that the support layer dominated the PV performance of the composite membrane when the PSF film evaporated in the air for more than 10 min.

The resistances in these membranes were shown in Fig. 9. In addition, it can also be observed from Fig. 9 that the overall resistance of the composite membrane was higher than the sum of the average resistance of top and support layer when the support showed a considerable resistance. Besides, due to the pores existing on the surface of the support layer, an interface layer would be produced unavoidably and therefore aggravated the inhomogeneity of the support structure, though the resistance of interface layer should be much smaller than PSF support with significant resistance in this study.

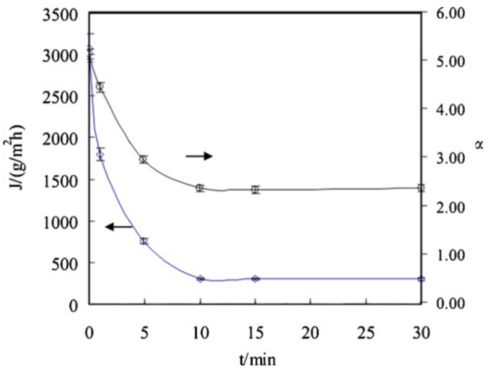

Fig. 8. Effect of evaporation time on PV performance of the PDMS–PSF membranes.

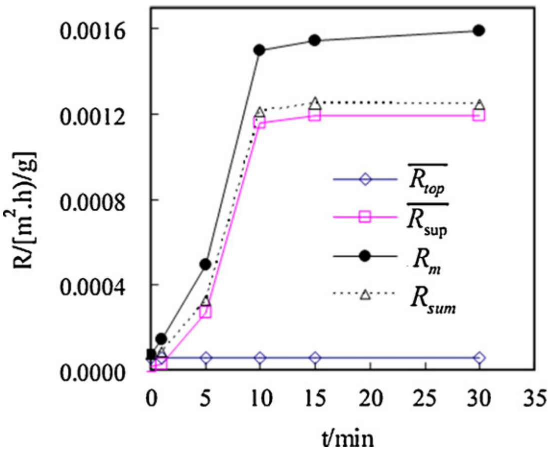

Fig. 9. The mass-transfer resistance in composite PDMS–PSF membranes.

5. Conclusions

From the above experimental results and discussions, the following conclusions can be drawn:

(1) With the increasing concentration of PSF in the casting solution and its evaporation time in the air, the resistance of PSF support layer would also increased and could dominate the PV performance of the composite membranes.
(2) The average parallel flow model is proposed to describe the influence of support layer structure on mass-transfer resistance in pervaporation. The Henis and Tripodi model can be included in the newly proposed model as a special case.
(3) When the support layer or any other interlayer in a composite membrane showed a considerable resistance, the inhomogeneous structure would produce the extra resistances in composite membrane.

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