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Pervaporation with Latex Membranes: A Study on Membrane and Pervaporation Effects

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ABSTRACT

The separation process is affected by a number of factors related to the membrane itself and the operation conditions. For latex membrane systems, factors such as feed concentration and feed temperature are found to affect pervaporation properties similarly to solvent-cast membrane systems. However, effects relating to polymer structure and latex particle morphology are more complicated for latex membrane systems. The separation performance depends upon the combined contribution of all the effects. It is shown that effects which make a latex membrane achieve a higher degree of film fusion, or make the polymer more rigid, will raise the membrane selectivity and decrease the permeation rate. A good latex membrane can be obtained by balancing all the effects pertaining to polymer, particle, and film structure and morphology.

INTRODUCTION

Pervaporation separation processes are affected by a number of factors relating to the membrane itself and the process control parameters. Studies on pervaporation with solvent-cast membranes indicate that the hydrophilic–hydrophobic balance of the membrane strongly affects the permeate–membrane interactions (1, 2). Copolymerization, grafting, crosslinking, and surface modification are effective methods to adjust this balance. Membrane morphology is also important, e.g., membrane separation factor may be greatly increased by increasing its degree of crystallinity (3).

Because latex membranes are formed differently from solvent-cast membranes (4), they have different effects. A typical effect of a latex membrane system is the change of pervaporation properties with its degree of film fusion. This effect, together with crystallinity and feed composition, were discussed in our previous papers (4, 5).

Permeate transport within a latex membrane during pervaporation is shown to be a combination of dissolution, diffusion, and desorption. Separation is achieved by selective sorption and selective diffusion. A simplified treatment was described previously in which the total permeation rate (J) is broken into the permeation rate through the polymer bulk (J_p) and the permeation rate through the boundary (hydrophilic) region (J_b).

$$J = J_p + J_b \tag{1}$$

where for any component i, the permeation rate is given by

$$J_i = J_{i,p} + J_{i,b} \tag{2}$$

Diffusion is considered to be the rate-determining step during pervaporation, and thus

$$J_i = -\left[(v_p)(D_{i,p})\frac{\partial C_{i,p}}{\partial x} + (v_b)(D_{i,b})\frac{\partial C_{i,b}}{\partial x}\right] \tag{3}$$

where v_p is the volume fraction of the polymer bulk, v_b is the volume fraction of the boundary region, and D is the diffusivity of permeant i in different regions. Subscripts "p" and "b" represent the polymer bulk and the boundary region, respectively.

Since the total permeation rate and membrane selectivity are results of the joint contribution of the permeation through polymer bulk and boundary region, factors affecting the permeation through either region would affect the latex membrane performance.

EXPERIMENTAL

Latex Preparation

Latexes used in this experiment are acrylonitrile–butyl acrylate copolymers. The basic emulsion polymerization recipe is:

Reactor charge:	
Distilled water	387 g
Sodium dodecyl sulfate (SDS)	1.5 g
Monomer mixture:	
Acrylonitrile	33.4 g
Butyl acrylate	64.8 g
EGDM (only for the crosslinked latex)	1.8 g

Initiator:	
Potassium persulfate (in 1.5 wt% aqueous solution)	0.2 g

The latex membrane compositions are given in the relevant figures and tables. The latexes were prepared by semicontinuous emulsion polymerization described previously (4). The core–shell latexes were prepared by starve feed of the core composition, followed by starve feed of the shell composition.

Membrane Preparation

Latex membranes are obtained by direct casting of the filtered latexes onto glass plates. The thicknesses of the membranes were between 80 and 120 μm.

After drying at ambient temperature in a moisture-controlled filtration hood for 7 days (unless otherwise specified), the membrane was carefully removed from the plate, followed by vacuum drying at ambient temperature for 24 hours to remove any volatile residuals.

Pervaporation

Pervaporation separation was conducted with ethanol–water mixtures. The apparatus and operation were described elsewhere (5).

Permeate composition was analyzed by a Perkin-Elmer 8310 Gas Chromatography with an FID detector. The column used was a 8′ × $\frac{1}{8}''$ SS packed with Hayesep P $\frac{60}{80}$ mesh. Nitrogen was used as the carrier gas at a flow rate of 20 mL/min.

The pervaporation separation performances of latex membranes are characterized by the permeation rate (J) and the separation factor (α), which is defined by Eq. (4):

$$\alpha_{(ij)} = \frac{Y_i/Y_j}{X_i/X_j} = \frac{Y_i/X_i}{Y_j/X_j} \tag{4}$$

where X and Y are ethanol weight fractions in the feed and permeate, respectively.

Scanning Electron Microscope (SEM)

The surface morphology of latex membranes was observed with a Hitachi S-570 Automatical Scanning Electron Microscope.

Latex Titration

NaOH solution was used to neutralize latexes containing acid groups.

Titration was indicated by pH values under constant stirring. pH values were measured by a Radiometer pH Meter 28.

Latex Particle Size and Size Distribution

Latex particle size and size distribution were measured by a ICI-Joyce Disk Centrifuge. The experimental data were recorded and processed by a Brookhaven Instruments DCP-1000 data system. The experimental measurement was described in Ref. 4. The result was represented in the form of number-average diameter (D_n) and polydispersity (D_w/D_n).

Dynamic Mechanical Analysis

Glass transition temperature (T_g), tan δ, and loss and storage moduli of the polymers were obtained using a Perkin-Elmer 7 Series Thermal Analysis System. A square-shaped film sample was placed in parallel plates. The measurement was conducted with a stress frequency of 1.00 Hz, and the rate of temperature scan was 5.0°C/min.

RESULTS AND DISCUSSION

Effect of Feed Temperature

For latex membranes, increasing feed temperature increases the permeation rate and decreases the separation factor, as shown in Fig. 1. Another effect, which is unique for the latex membrane system, is the permeation rate reduction with increasing feed temperature. This effect was discussed in another paper (5). For some latex membrane systems, the temperature effect can be described by Arrhenius-type relationships (5). It was also shown that the change in separation properties of latex membranes with feed temperature was significantly affected by the T_g of the polymer (5).

Effect of Latex Membrane Casting Temperature

A continuous latex film is formed only if the latex is dried above its minimum film temperature (MFT) (4). Above the MFT, increasing membrane casting temperature is equivalent to increasing membrane aging time or aging temperature, since a higher casting temperature enables latex particles to achieve better fusion and a larger extent of polymer chain segment interdiffusion. The boundary region also becomes less distinct. The effect of membrane casting temperature is presented in Fig. 2.

As seen from Fig. 2, both separation factor and permeation rate change significantly around a specific temperature, which happens to be the mem-

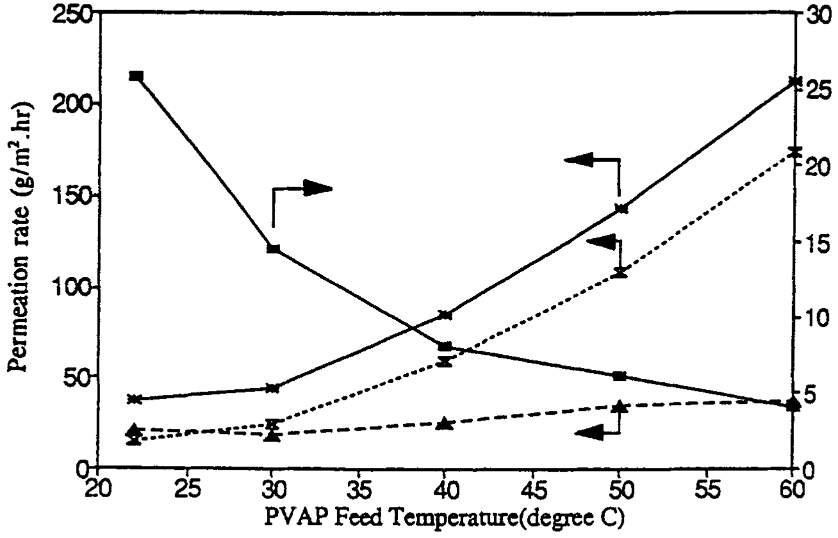

FIG. 1 Effect of feed temperature on permeation rate and selectivity; membrane: cross-linked P(AN–BuA); feed concentration: 95 wt% ethanol. (■) Separation factor; (*) total permeation rate; (X) ethanol permeation rate; (▲) water permeation rate.

brane T_g (36.5°C) (4). The reduction in permeation rate near this temperature is due to the increase in the degree of film fusion. When the casting temperature is below the polymer T_g, the latex film is poorly fused, and thus the permeation rate is high and the separation factor is low. When the casting temperature is higher than the T_g, polymer chains have high mobility and chain interdiffusion at the particle–particle interface, which results in decreasing J_p and J_b. Further increasing casting temperature does not significantly increase the degree of film fusion, thus permeation rate and separation factor are relatively constant.

Effect of Amount of Acrylonitrile in Copolymer

Acrylonitrile(AN) has a strong polar group (—CN) in its molecule. Solvent-cast membranes made of acrylonitrile homopolymer and copolymers were reported to have good selectivity toward water in the pervaporation separation of ethanol–water mixtures (6, 7). Since poly(acrylonitrile) (PAN) latex film is too brittle, butyl acrylate was incorporated to improve the film formation of PAN. However, the introduction of butyl acrylate has a negative effect on water selectivity of PAN membranes. Previous

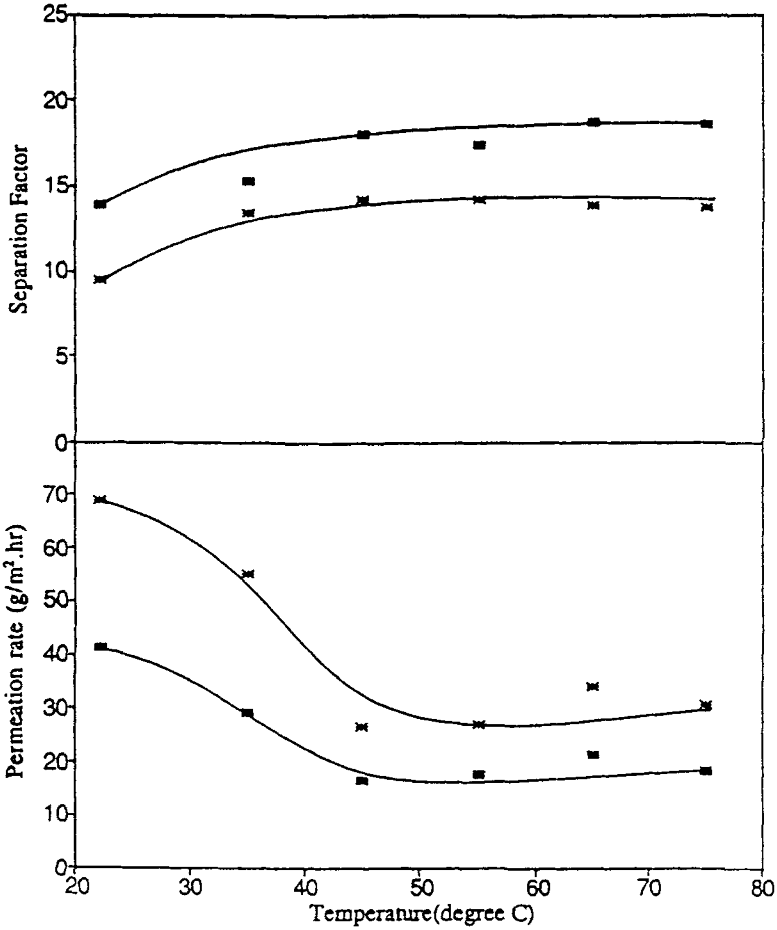

FIG. 2 Effect of membrane casting temperature on permeation rate and selectivity; membrane: crosslinked P(AN–BuA); drying time: 48 hours at the casting temperature; feed concentration: 95 wt% ethanol; feed temperature: (■) 25°C, (*) 35°C.

study showed that some solvent-cast polyacrylate membranes are selective to organic compounds in pervaporation (8, 9).

Figure 3 gives pervaporation properties as a function of polymer composition. Increasing acrylonitrile content apparently increases separation factor due to a larger fraction of the strong polar groups in the membrane and a higher degree of crystallinity. This effect can be explained according

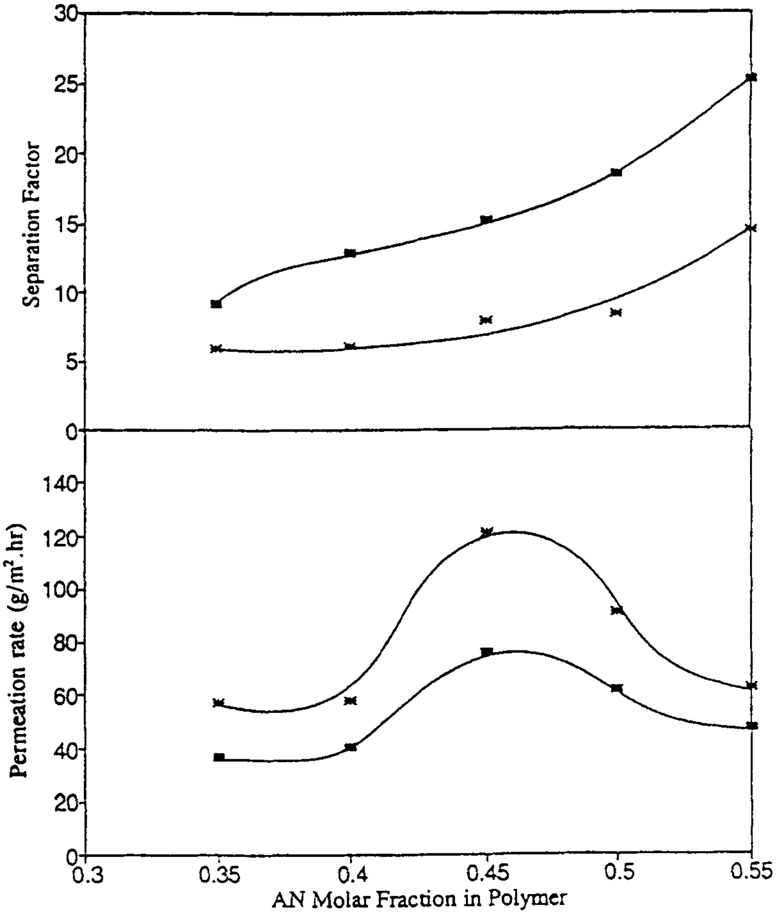

FIG. 3 Effect of copolymer composition on pervaporation properties; feed concentration: 95 wt% ethanol; feed temperature: (■) 25°C, (*) 35°C.

to the transport mechanism through latex membranes which was presented earlier (5).

Table 1 gives the glass transition temperatures of polymers with different AN fractions. Data in Table 1 indicate that a polymer with a higher AN content has a higher T_g.

When membranes having different T_gs are cast at the same temperature and applied to the same aging time, the one with a higher AN content

TABLE 1
T_g for Copolymers with Different AN Contents

AN molar fraction	0.35	0.4	0.45	0.50	0.55
T_g (°C)	0.0	8.9	17.9	29.1	36.5

(thus with higher T_g and more rigid) would have a lower degree of film fusion, leading to a higher J_b. On the other hand, due to its higher degree of crystallinity, a polymer with higher AN content is less permeable to liquids, which results in a lower J_p. The joint contribution of these two effects determines the total permeation rate which eventually exhibits a bell shape with changing polymer composition. In such a system, if AN is simply viewed as the hydrophilic component and BuA as the hydrophobic one, the above results suggest that hydrophilic–hydrophobic balance is important.

Effect of Crosslinking Density

To use latex films in membrane separation processes, crosslinking is often necessary. If no chemical crosslinks are introduced into the latex membrane, it may be swollen extensively by feed mixtures, especially at high organic concentrations.

Introduction of crosslinks makes the polymer chains more three-dimensional and less mobile, so that the degree of membrane swelling and permeant diffusion are limited. Increasing the degree of crosslinking decreases the polymer chain mobility, as indicated by their glass transition temperatures listed in Table 2.

Generally, increasing the degree of crosslinking has two effects on a latex membrane during pervaporation. 1) It leads to a more compact polymer structure and causes higher selective diffusion through the polymer bulk, which tends to increase the separation factor. This also deceases the permeation rate through the polymer bulk, J_p. 2) Increasing the degree of crosslinking makes latex particles more rigid, reducing the rate of mutual interdiffusion of polymer chain ends in the boundary region, thus

TABLE 2
Change in T_g and Water Sorption as a Function of Polymer Crosslinking Density

EGDM (wt fraction)	0.00	0.03	0.06	0.08
T_g (°C)	14.4	22.8	25.8	33.1
Water sorption (g/g)	0.24	0.20	0.16	0.14

reducing the rate of particle coalescence (4), preventing latex particles from achieving good fusion. This does not benefit the selective diffusion through the boundary region, thus decreasing the separation factor. A poorly fused film also causes large permeation through the boundary region, J_b.

As a result of the joint contribution of the above two effects, increasing the degree of crosslinking does not always improve the pervaporation separation properties of latex membranes. Figure 4 shows permeation rate and separation factor as functions of crosslinking agent concentration. Since the crosslinks were introduced by copolymerization with the difunctional monomer ethylene glycol dimethyl acrylate (EGDM), increasing its concentration is equivalent to increasing the crosslinking density. In Fig. 4 it is shown that a significant increase in separation factor is obtained only when a large amount of crosslinking agent is incorporated, while a continuous decrease in permeation rate is observed with increasing the degree of crosslinking.

Effect of Latex Particle Size and Size Distribution

Based on the proposed pervaporation transport mechanism through latex membranes which is shown by Eq. (3), one of the factors determining the total permeation rate is the volume fraction of polymer bulk (v_p) and boundary region (v_b), which are functions of particle size and size distribution. Theoretically, with the same membrane thickness (thus the same membrane volume), the larger the particle size, the smaller the total particle surface area, i.e., a smaller v_b, which leads to a lower J_b. The opposite effect is that larger latex particles have lower degrees of film fusion at the same aging time and temperature, as shown in Fig. 5, which would increase permeation through the boundary region, J_b. Again, the pervaporation properties are determined by the contribution of these two effects.

In Table 3, pervaporation properties of latex membranes with different particle sizes and size distributions are listed. As can be found from Table 3, the total permeation rate increases with increasing particle size. If J_p is assumed constant with different particle sizes, this result suggests that the effect of film fusion on permeation rate (J) is more important than the volume effect caused by the change in particle size. As expected, the lowest selectivity is found with the largest particle size, due to its lowest degree of membrane fusion.

Effect of Surfactant Concentration

The emulsifier, which is a surfactant and contains hydrophilic and hydrophobic groups, serves as a particle stabilizer during latex preparation.

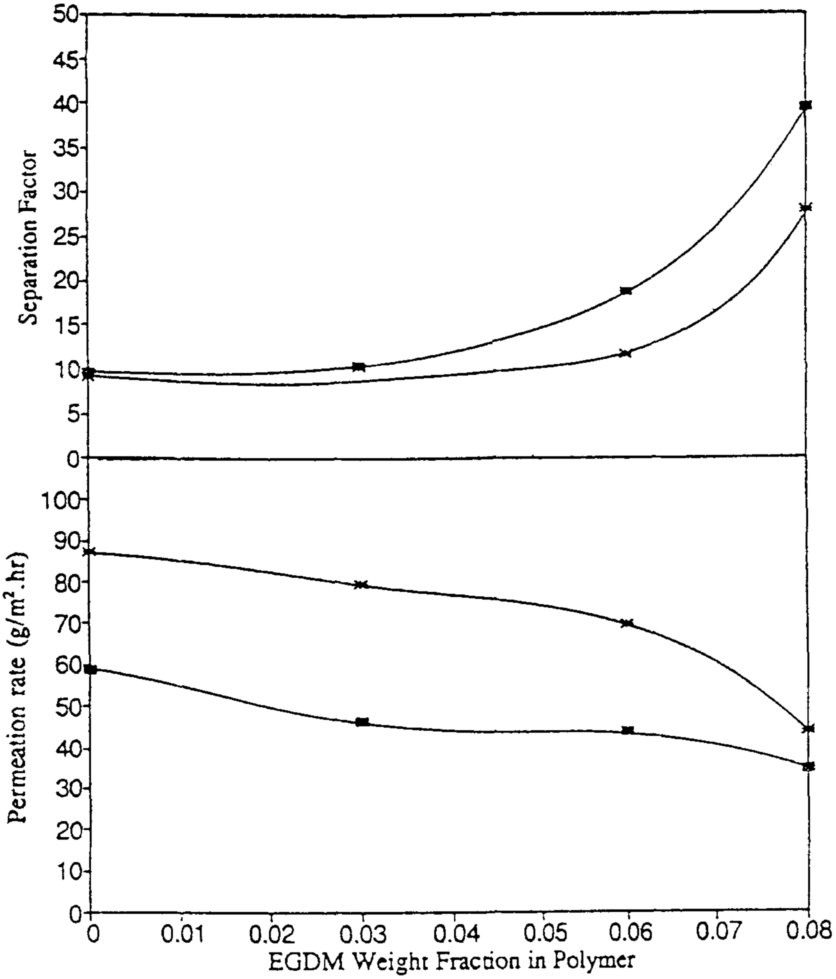

FIG. 4 Effect of degree of crosslinking on permeation rate and separation factor; feed concentration: 95 wt% ethanol; feed temperature: (■) 25°C, (*) 35°C.

The surfactant molecules are not involved in the chemical reaction, and they remain bounded or unbounded on the surface of the particles. Surfactant is one of the major compositions of the boundary region (except for soap-free latex membranes).

Increasing emulsifier concentration in emulsion polymerization has two effects on the properties of the resulting latex membrane. 1) It increases

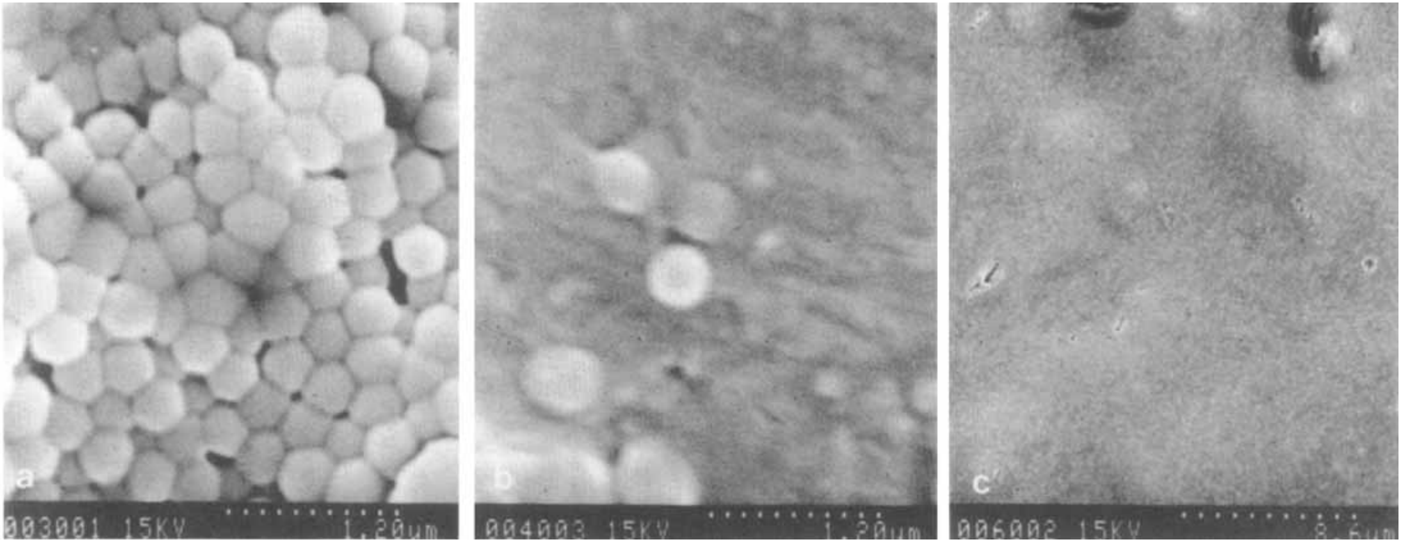

FIG. 5 Scanning electron micrographs of latex films; latex: crosslinked P(AN–BuA); casting temperature: 20°C; drying time: 6 hours; particle size: (a) 392.5 nm, (b) 143.8 nm, (c) 103 nm.

TABLE 3
Effect of Particle Size and Size Distribution on Pervaporation Properties. Feed Temperature, 25°C; Ethanol Concentration, 95 wt%

D_n (nm)	D_w/D_n	J (g/m^2·h)	α
103.0	1.11	17.12	17.99
143.8	1.10	32.88	18.92
392.5	1.00	48.16	15.97

the surfactant concentration in the latex membrane, which makes the boundary region more hydrophilic due to its strong interaction with water. This effect is referred to as the *interaction effect*. 2) It reduces latex particle size, and thus increases the degree of film fusion, which is referred to as the *particle size effect*. As discussed earlier, the first effect tends to cause a higher permeation through the boundary region J_b while the second effect decreases J_b. The overall contribution determines the total permeation rate and separation factor. Here it is assumed that permeation through the polymer bulk is not affected by the surfactant concentration, since changing emulsifier concentration alone does not significantly change polymer structure and particle morphology.

The effects of emulsifier concentration on permeation rate and separation factor are shown in Fig. 6. As can be seen from Fig. 6(a), the separation factor does not change significantly with increasing emulsifier concentration, suggesting that the two effects on membrane selectivity are canceled out.

The effect of emulsifier concentration on the permeation rate (J) is significant. According to the above discussion, the total permeation rate can be used to indicate the change in the permeation through the boundary region (J_b), as shown in the following.

1. J of the membrane with zero emulsifier (via soap-free emulsion polymerization) is lower than the one with 0.15 weight fraction emulsifier (based on monomer amount). This suggests that when a small amount of emulsifier is introduced into the system, the interaction effect is more significant in determining J_b than the particle size effect, which increases J.
2. When the emulsifier concentration in the system is large enough, further increasing its concentration causes a reduction in J, thus in J_b, which shows that the particle size effect becomes more important in affecting J_b within this range.
3. When the emulsifier concentration is higher than about 0.03, the parti-

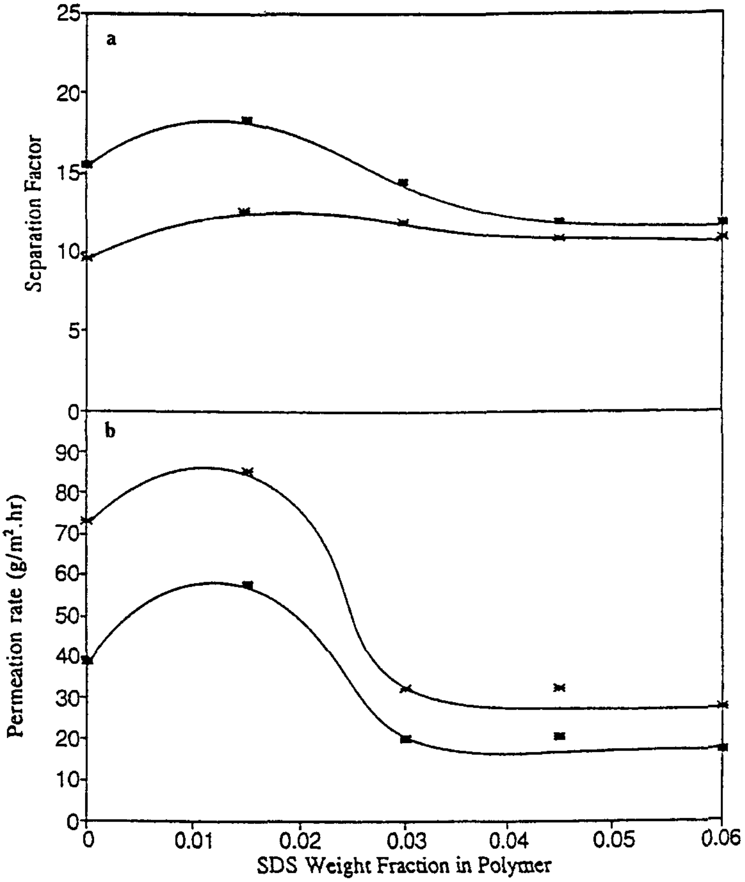

FIG. 6 Effect of surfactant concentration on permeation rate and separation factor; feed concentration: 95 wt% ethanol; feed temperature: (■) 25°C, (*) 35°C.

cle size will not continue to be reduced significantly; the permeation rate eventually reaches a constant value within the experimental range.

4. Comparing the J at an emulsifier concentration higher than 0.04 with J of an emulsifier-free latex membrane, the latter is significantly higher. It is therefore concluded that the particle size effect, which is related to the degree of membrane fusion, is more important in determining the boundary region permeation, J_b, than the interaction between surfactant and permeant.

It should be noted, however, that the surfactant was introduced into the reactor at the beginning of emulsion polymerization, therefore these molecules are basically bounded to latex particles. Also, no surfactant extrusion was observed by SEM (4). In a comparison experiment, the same amount of surfactant was added to the soap-free latex, then the latex was cast and dried under the same conditions. Such films exhibited apparent surfactant extrusion and film shrinking, and could not be used for pervaporation tests. Consequently, the above discussion on the effects of surfactant concentration only applies to systems where the surfactant molecules are bound to latex particles or completely miscible with the polymer bulk.

Effect of Latex Particle Morphology

Particle morphology largely determines the latex membrane morphology and its pervaporation properties. If the membrane is prepared from copolymers, different copolymer structures or latex particle morphologies can be produced, depending on how the monomers are fed to the reactor. Even with starve feed, no perfectly uniform particle morphology can be obtained because the copolymer composition drift cannot be reduced to zero (due to mass and temperature flocculation, reflux, reactivity ratios, etc.).

Morphologically heterogeneous latex particles are often prepared through multistage emulsion polymerization, where different monomer or comonomers are fed at different stages. A number of different morphologies have been reported (10, 11). A typical heterogeneous particle structure is the "core–shell" morphology. Such a structure can be produced through two-stage polymerization where a second stage monomer (or monomers) is fed to the reactor containing a preformed seed. However, a two-stage process does not always generate the desired core–shell morphology (10).

A core–shell latex was used to study the effect of latex particle morphology on pervaporation properties. In Fig. 7 the loss modulus of the polymer is plotted as a function of temperature. It is seen from this figure that these membranes exhibit different glass transitions, indicating their differences in structures and the formation of a core–shell morphology. Curve (a) in Fig. 7 shows a single transition, which is the copolymer T_g. In Curve (c) the two peaks correspond to the T_g of the homopolymers, symbolizing a core–shell structure is formed when AN is added at the second stage, with poly(butyl acrylate) as the core and polyacrylonitrile as the shell. However, when AN is fed first, a certain degree of phase inversion occurs due to the strong hydrophilicity of acrylonitrile. As shown by Curve (b),

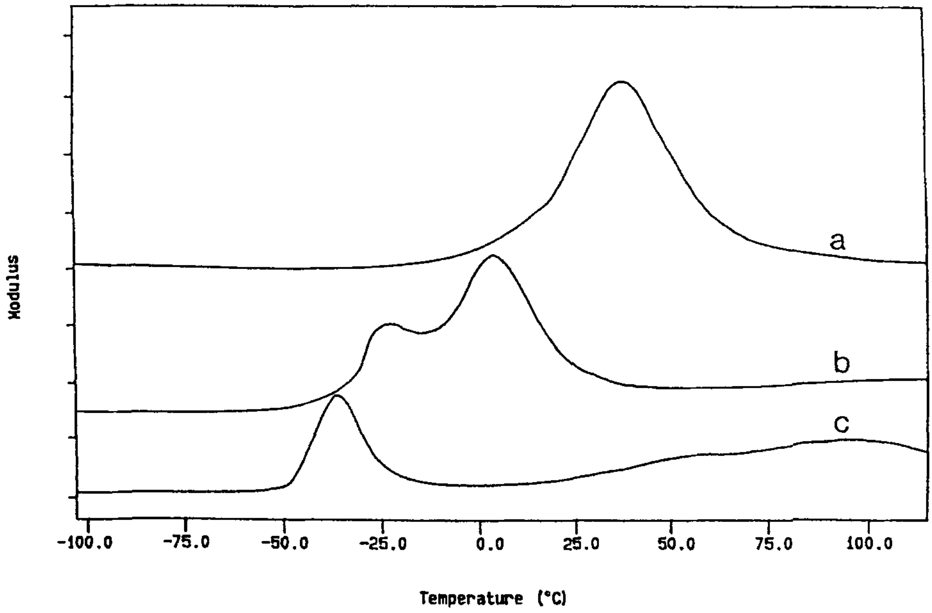

FIG. 7 Loss moduli of emulsion polymers having different morphologies. (a) AN–BuA copolymer; (b) AN core, BuA shell; (c) BuA core, AN shell.

instead of a true core–shell structure, particles having a two-phase morphology are produced, possibly with one phase rich in BuA while the other is rich in AN. The permeation rate and selectivity of membranes having different particle morphologies are listed in Table 4.

From the results in Table 4 it is seen that different particle morphologies lead to different pervaporation properties.

TABLE 4

Effect of Latex Particle Morphology on Pervaporation Properties. Feed Temperature, 25°C; Ethanol Concentration in Feed, 95 wt%

Sample	First stage monomer[a]	Second stage monomer[a]	Particle morphology	J (g/m^2·h)	α
1	AN + BuA	AN + BuA	Homogeneous	29.0	49.84
2	AN	BuA	BuA shell	34.9	8.71
3	BuA	AN	AN shell	61.6	7.51

[a] 2 wt% EGDM (based on monomer weight) is added together with the monomer.

1. In Table 4, Sample 1 is made of AN–BuA copolymer, while Samples 2 and 3 are primarily made of homopolymers (there may be some copolymerization or grafting). Although their polymerization recipes are the same, the pervaporation properties of these latex membranes are different, with the copolymer latex membrane having a higher selectivity and lower permeation rate. However, it is not implied that a homogeneous copolymer has to be superior to a blend of the corresponding homopolymers. In this case, a uniform latex particle structure benefits the separation.
2. Comparing the permeation rate and selectivity of Samples 2 and 3, it is found that if AN is the shell composition, the latex membrane shows a higher permeation rate and a lower selectivity. Because a rigid shell rich in PAN makes the deformation of the particles difficult, and the interdiffusion of the polymer chain ends at the particle–particle interface hard, film fusion is poor. As a result, the total permeation rate is high and selectivity is low. Besides, the difference in the particle surface polarity due to different shell composition may also change selective diffusion through the boundary region.

Effect of Hydrophilic–Hydrophobic Balance of Latex Membranes

The hydrophilic–hydrophobic balance of the copolymer is changed by adding acrylic acid (AA) or methyl acrylic acid (MAA) as the comonomer. Data in Table 5 show that membranes with carboxyl groups in the polymer have higher permeation rates. But a reduction in selectivity is observed. A possible reason is that when the hydrophilic group (—COOH) is introduced into the polymer, greater swelling of the membrane happens due to increasing interaction with the feed mixture, which also benefits the permeation of ethanol.

To demonstrate the effect of boundary region hydrophilicity on membrane properties, an experiment was conducted in which the polarity of

TABLE 5
Effect of Introducing —COOH on Pervaporation Properties. Feed Temperature, 25°C; Ethanol Concentration in Feed, 95 wt%

Latex membrane	[—COOH] (meq/g polymer)	J (g/m^2·h)	α
AN–BuA	0.0	39.3	25.1
AN–BuA–AA	0.2775	45.1	13.6
AN–BuA–MAA	0.2775	57.5	10.2

the boundary region was adjusted by changing —COOH to —COONa. To obtain the samples, the AN–BuA–AA copolymer latex was titrated with NaOH to reach different pH values, then it was cast onto a glass plate and dried in the same way as described earlier. As shown by the titration curve in Fig. 8, the end point is around pH 8.5. Thus, below this point the —COONa concentration in the membrane is proportional to the amount of NaOH added to the system. The concentration of —COONa in the solid membrane is calculated based on Eq. (5):

$$[\text{—COONa}] = VM/wq \tag{5}$$

where [—COONa] is in milliequivalents, V is the volume of NaOH solution consumed (in mL), M is the molarity of the NaOH solution, w is the weight of the latex (in g), and q is the solid content of the latex.

Table 6 gives water sorption as a function of —COONa concentration. It is shown that water uptake increases with increasing [—COONa] due to the greater interaction between membrane and water, which also indicates an increase in the degree of membrane swelling.

Pervaporation with membranes having different —COONa concentrations was conducted, and the results are presented in Fig. 9 which shows a higher permeation rate at higher [—COONa], with a slight decrease in selectivity. Again, the increase in the degree of membrane swelling caused

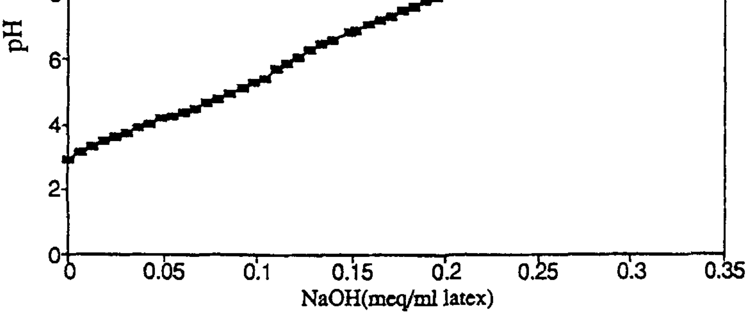

FIG. 8 Latex titration curve.

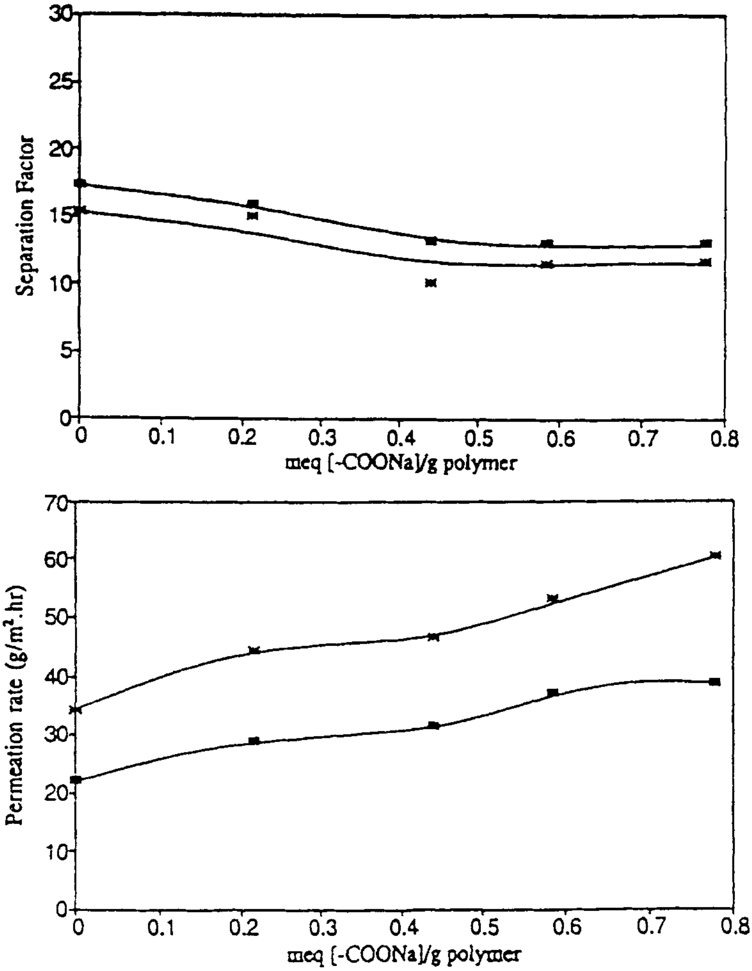

FIG. 9 Effect of [—COONa] on pervaporation properties; feed concentration: 95 wt% ethanol; feed temperature: (■) 25°C, (*) 35°C.

TABLE 6
Effect of —COONa Concentration on Membrane Water Sorption

[—COONa] (meq/g membrane)	Water sorption (g/g membrane)
0.00	0.32
0.21	0.58
0.43	0.71
0.59	0.82
0.79	0.91

by increasing [—COONa] is responsible for this effect. It is suggested by this observation that changing the boundary nature will change pervaporation properties of the latex membrane.

CONCLUSIONS

Factors related to process control, such as feed concentration and feed temperature, affect pervaporation of a latex membrane in a way similar to solvent-cast membrane systems. It is demonstrated that effects become more complicated when factors related to the emulsion polymer structures and latex particle morphology are considered.

Factors affecting latex membrane properties can be further divided into three categories. The first are factors pertaining to the polymer structure, such as polymer composition, chain regularity and polarity, crosslinking, and crystallinity. The second are those aspects which mainly alter particle morphology, e.g., batch or semicontinuous polymerization process, emulsifier system. The third group includes conditions which influence the membrane (latex film) morphology, such as aging time, thermal treatment, and different aging medium. The current study reveals that changing one parameter usually results in more than one effect on the polymer bulk and boundary region. For example, increasing crosslinking density leads to a denser polymer bulk, which will reduce J_p and raise selectivity; at the mean time it prevents the latex particles from achieving better fusion, which will raise J_b and reduce selectivity. The overall performance of such a system depends upon the combined contribution of these effects, thus J and α may either be increased or decreased.

It is observed that factors which make a latex membrane fuse better, or make the polymer more rigid, would raise the membrane selectivity but decrease its permeation rate. A good latex membrane can only be obtained if all the effects pertaining to polymer, particle, and film structures and morphologies are properly controlled.

NOTATIONS

c	concentration
D	diffusion coefficient
D_n	number-average particle diameter
D_w	weight-average particle diameter
D_w/D_n	polydispersity of particle size
J	permeation rate
J_b	permeation rate through the boundary region
J_p	permeation rate through the polymer bulk
M	molarity
q	solid content of latex
T_g	glass transition temperature
T	temperature in Kelvin scale
V	latex volume
v_b	volume fraction of boundary region
v_p	volume fraction of polymer bulk
w	latex weight
x	membrane thickness
X	weight fraction in feed
Y	weight fraction in permeate
α	separation factor

Subscript

b	boundary
i, j	type of feed component
m	membrane
p	polymer

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