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Removal of Acetic Acid from Wastewater with Liquid Surfactant Membranes: An External Boundary Layer and Membrane Diffusion Controlled Model

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ABSTRACT

A new diffusion controlled mass transfer model has been established for uniform emulsion globules without redispersion and internal circulation. Both the external boundary layer and the membrane resistance to mass transfer are taken into account and the perturbation solutions have been obtained. Experimental data on the batch extraction of acetic acid from wastewater are in good agreement with the model predictions. Usually, the zero-order, or pseudo-steady state solution alone can give an adequate description of the process.

The swell of emulsion can be classified into two types - permeation and entrainment. The swelling rate by permeation of water increases with increasing the concentration of surfactant and internal reagent. Repeated redispersion and vigorous stirring promote the swell due to the entrainment. The swelling mechanism of the entrainment has been discussed.

INTRODUCTION

The liquid surfactant membranes (LSM) as a novel separation technique invented by Dr. Li (1) have been widely used in many fields such as the fractionation of hydrocarbons, environmental engineering, hydrometallurgy, nuclear industry, pharmaceutical and biological engineering (2-7). Recently, the LSM has demonstrated considerable potential as effective tools for the removal of trace contaminant from industrial effluents. In present paper, the removal of acetic acid from wastewater was tested and special attention has been paid to the fundamental studies.

From the recent literature, it is found that the mass transfer models in the LSM can be classified into two distinct types. The first is the spherical shell model in which the mass transfer resistance was assumed to be concentrated within a film of fixed thickness (2,8). The second is called the Advancing Front Model that deemed the membrane resistance to be a continuous function of extraction time (9-12). But mass transfer resistance in the boundary layer of external phase was not considered in either model and might be important at the beginning of extraction or at the state when the concentration of internal reagent was high. Teramoto et al (13) established a new model by including the resistance in the boundary layer of the external phase and the chemical equilibrium, but only numerical solutions can be obtained from such complex equations. In this paper, by taking both the resistance in external boundary layer and membrane phase into account and modifying the Advancing Front Model, a new mass transfer model has been developed. Furthermore, the perturbation solutions are obtained and the model predictions can be examined by the experimental data on the extraction of acetic acid from wastewater.

MASS TRANSFER MODEL

The assumptions applied for establishing the model are given as follows:

1. The size distribution of emulsion globules is uniform. No coalescence and redispersion occur between the globules in which the encapsulated droplets are uniformly distributed.

2. There is no internal circulation within all emulsion globules due to the presence of surfactants and the small dimension of the globules.

3. The solute reacts with the internal reagent irreversibly and instantaneously at the reaction front. As the reaction proceeds, the reaction front shrinks towards the core of the globules.

4. Both the important effects of resistance in the external boundary layer and the membrane are taken into account in the model.

5. The breakage and the swell of the emulsion are neglected.

A diagram of the model showing the above assumptions is given in Fig. 1.

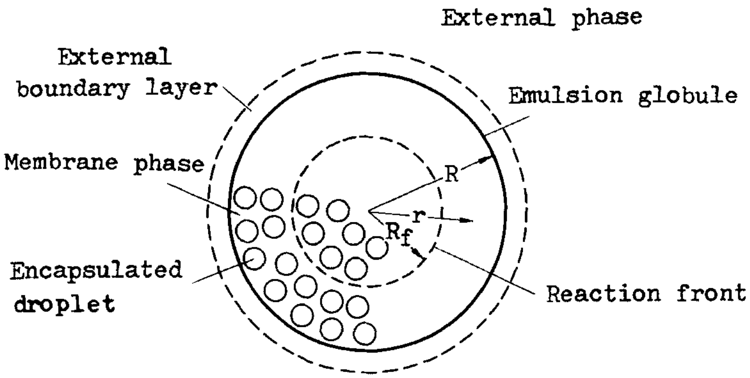


Fig. 1 Schematic diagram of the model

The rate of the solute diffusion in emulsion globules can be described in the following equations:

$$\frac{\partial C}{\partial t} = \frac{D_e}{r^2}\frac{\partial}{\partial r}\left(r^2\frac{\partial C}{\partial r}\right) \qquad (R_f(t) < r < R,\ t > 0) \qquad (1)$$

$$t = 0, \qquad C = 0 \qquad (r \leqslant R) \qquad (2)$$

$$r = R_f(t), \qquad C = 0 \qquad (t \geqslant 0) \qquad (3)$$

$$r = R, \quad D_e\frac{\partial C}{\partial r}\bigg|_{r=R} = K\left(C_e - \frac{C}{\alpha}\bigg|_{r=R}\right) \qquad (t \geqslant 0) \qquad (4)$$

The material balance for the solute in the external phase is

$$-V_e\frac{dC_e}{dt} = \frac{3}{R}(V_i + V_m)\, D_e\frac{\partial C}{\partial r}\bigg|_{r=R} \qquad (5)$$

$$t = 0, \qquad C_e = C_{eo} \qquad (6)$$

The material balance for the solute at the reaction front is

$$-\left(\frac{V_i}{V_i+V_m}\right)C_{io}\frac{dR_f}{dt} = D_e\frac{\partial C}{\partial r}\bigg|_{r=R_f(t)} \qquad (7)$$

$$t = 0, \qquad R_f = R \qquad (8)$$

The above equations can be transformed into dimensionless form by defining

$$\eta = \frac{r}{R}, \quad x = \frac{R_f}{R}, \quad \tau = \frac{\varepsilon D_e t}{R^2}, \quad g = \frac{C}{\alpha C_{eo}}, \quad h = \frac{C_e}{C_{eo}},$$

$$\varepsilon = \frac{\alpha(V_{io}+V_m)C_{eo}}{V_{io}C_{io}}, \quad E = \frac{3C_{io}V_{io}}{C_{eo}V_{eo}}, \quad M = \frac{D_e}{KR} \qquad (9)$$

then the diffusion equation becomes

$$\varepsilon \frac{\partial g}{\partial \tau} = \frac{1}{\eta^2} \frac{\partial}{\partial \eta}(\eta^2 \frac{\partial g}{\partial \eta}) \qquad (x < \eta < 1, \ \tau > 0) \qquad (10)$$

$$\tau = 0, \qquad g = 0 \qquad (\eta \leqslant 1) \qquad (11)$$

$$\eta = x, \qquad g = 0 \qquad (\tau \geqslant 0) \qquad (12)$$

$$\eta = 1, \qquad \left.\frac{\partial g}{\partial \eta}\right|_{\eta=1} = \frac{1}{\alpha M}(h - g\big|_{\eta=1}) \qquad (\tau \geqslant 0) \qquad (13)$$

The material balance equation in the external phase is

$$\frac{dh}{d\tau} = -E \left.\frac{\partial g}{\partial \eta}\right|_{\eta=1} \qquad (14)$$

$$\tau = 0, \qquad h = 1 \qquad (15)$$

The material balance equation at the reaction front is as follows:

$$\frac{dx}{d\tau} = - \left.\frac{\partial g}{\partial \eta}\right|_{\eta=x(\tau)} \qquad (16)$$

$$\tau = 0, \qquad x = 1 \qquad (17)$$

Because these equations are non-linear, analytical solutions can not be obtained. However, by using the perturbation method used in the Advancing Front Model (11), the zero-order solutions are found to be

$$h_o = \frac{E}{3}(x^3 - B^3) \qquad (18)$$

$$\tau_o = \frac{1}{E}\left\{(1 + \frac{1}{2B} - M\alpha) \ln\frac{x^3-B^3}{1-B^3} - \frac{3}{2B} \ln\frac{x-B}{1-B} - \frac{\sqrt{3}}{B}\left[tg^{-1} \frac{2x+B}{\sqrt{3}\,B} - tg^{-1} \frac{2+B}{\sqrt{3}\,B}\right]\right\} \qquad (19)$$

where

$$B = (1 - \frac{3}{E})^{1/3} \tag{20}$$

The first-order terms of the perturbation solution can be obtained as indicated below:

$$\left\{\begin{array}{l} \frac{dh_1}{dx} = \frac{E^2L^2}{18}p^2(1-x)^2(x^3-B^3) - \frac{E^2L(L-1)}{18}p^2x(1-x)^2(x^3-B^3) \\ \quad - \frac{E^2L}{6}px(1-x)(x^3-B^3) + \left[\frac{EL}{2}p^2x(1-x) - \frac{EL^2}{3}p^3(1-x)^2 \right. \\ \quad \left. + \frac{EL(L-1)}{3}p^3x(1-x)^2\right]\left[Q + \frac{E}{3}(x^3-B^3)\right] \qquad (21) \\ x = 1, \quad h_1 = 0 \qquad (22) \end{array}\right.$$

$$\left\{\begin{array}{l} \frac{d\tau_1}{dx} = \frac{x-1}{2} + \frac{P}{2}(1-x) - \frac{L-1}{6}p(1-x)^2 + \frac{L-1}{3}p^2(1-x)^2 \\ \quad - \frac{xh_1}{Lph_o^2} + Q\left[\frac{1-x}{2h_o}p + \frac{L-1}{3h_o}p^2(1-x)^2\right] \qquad (23) \\ x = 1, \quad \tau_1 = 0 \qquad (24) \end{array}\right.$$

where

$$L = \frac{1}{\alpha M}, \qquad P = \frac{1}{(L-1)x-L} \tag{25}$$

$$Q = E(2L-1)x^3 - E(L-1)x^4 - ELx^2 \tag{26}$$

then, the first-order perturbation solutions are

$$h^{(1)} = h_o + \varepsilon h_1 \tag{27}$$

$$\tau^{(1)} = \tau_o + \varepsilon\tau_1 \tag{28}$$

where ε is a perturbation parameter. The above solutions can be simplified for $E=3(B=0)$ or $L=1$.

The parameter M in the above solutions represents the ratio of mass transfer resistance in the external boundary layer to the maximum resistance in the emulsion globules. If M equals zero, the solutions are the same as those of the Advancing Front Model. Because M and other parameters can be calculated or estimated by using suitable correlations, this model has more advantages over those of the Advancing Front Model.

The effect of the perturbation parameter ε on the calculated value of dimensionless concentration in the external phase is shown in Fig. 2. It is found that h_0 and $h^{(1)}$ are convergent. When E=2 and $\varepsilon < 0.2$, the deviations between h_0 and $h^{(1)}$ are probably within the bounds of experimental error. The tolerant value of ε at which $h^{(1)}$ can be replaced by h_0 increases with increasing the value of E. Usually, during the process of liquid membrane separations, reagent concentration in the internal phase is higher while solute concentration in the external phase is lower, so that larger E and smaller ε values occur in the process. Therefore, a zero-order, or pseudo-steady state solution is adequate to describe the mass transfer process.

The effect of the value of E on mass transfer rate is shown in Fig. 3. The larger the value of E, the faster the mass transfer rate of the diffusing component, but the effect falls off when E increases to a certain value.

Fig. 4 shows the effect of M on mass transfer rate. The mass transfer rate decreases with the increase in M, which results from the increase in the mass transfer resistance in the boundary layer of the external phase.

EXPERIMENTAL

In order to prepare water in oil (W/O) emulsion, the substances were screened as follows:

Emulsifier - nonionic polyamine E644
Carrier - TBP
Membrane solvent - kerosene and liquid paraffine
Internal reagent - NaOH

Typical ingredients of emulsion are (by weight): 8% E644, 72% kerosene, 19% liquid paraffine, 1% TBP and 3M NaOH.

The W/O emulsion was prepared as follows. At first, the mixed membrane solution of 100 ml was introduced into a stirrer of ϕ 60 mm, h 90 mm, with four baffles and two six-blade turbines, then at the speed of 1,500 rpm, NaOH solution of 50 ml was added into the stirrer. After stirring for 20 minutes, W/O emulsion was obtained.

Mass transfer experiments were carried out in a stirrer of ϕ 90 mm, h 150 mm, with a six-blade turbine and a thermostatic water jacket. A 0.0861M acetic acid solution of 405 ml was first placed in the stirrer, then the above mentioned emulsion of 135 ml was dispersed in the solution by agitation. Samples for analyzing acetic acid concentration in the external phase were periodically removed from the bottom of the stirrer and analyzed by titration. The temperature was 298 K.

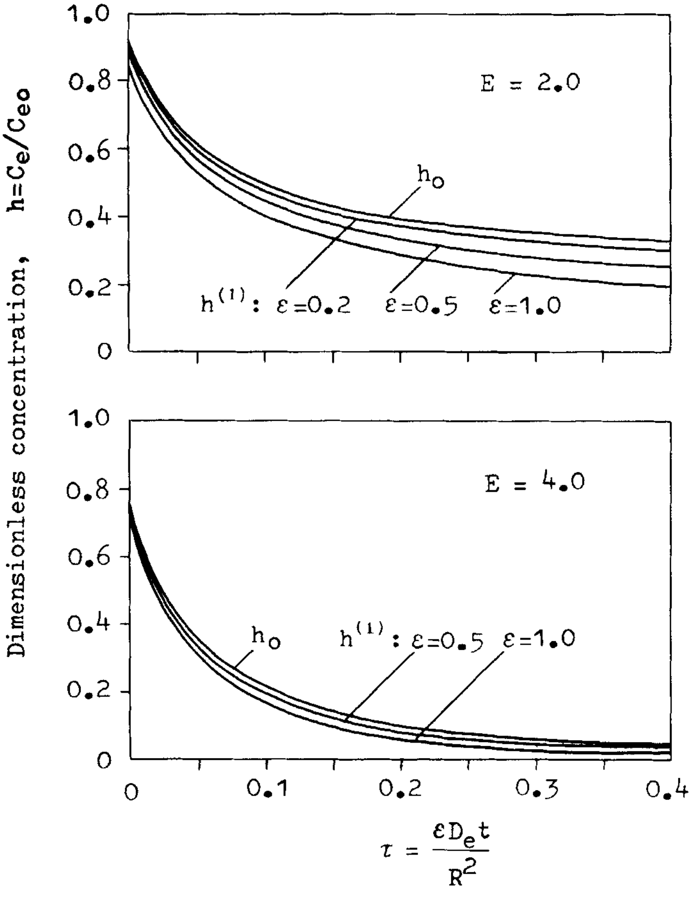


Fig. 2 The effect of ε on the dimensionless external phase concentration (M=0.1)

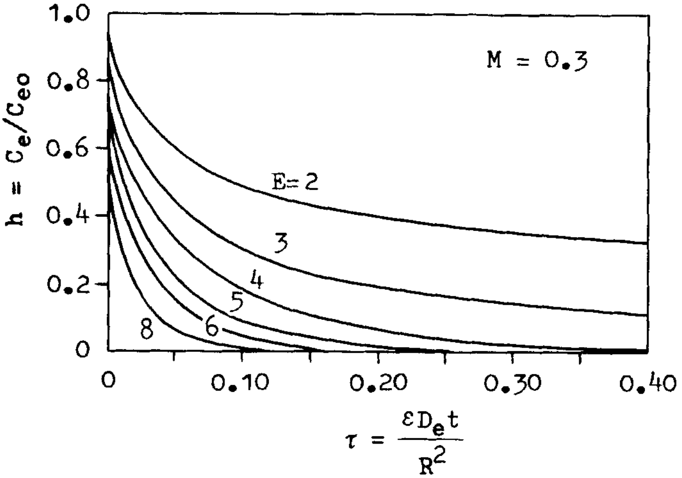


Fig. 3 The effect of E on the dimensionless external phase concentration

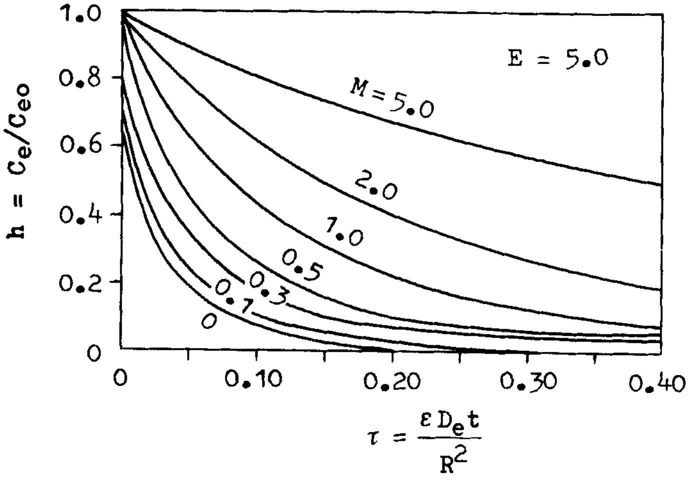


Fig. 4 The effect of M on the dimensionless external phase concentration

EXAMINATION OF THE MASS TRANSFER MODEL

In order to compare the experimental data with model predictions, the mass transfer coefficient, K, of acetic acid in the boundary layer of the external phase, the distribution coefficient, α', between the external and membrane phase, the effective diffusion coefficient, D_e, of acetic acid in the saturated zone of the emulsion globule, and the mean diameter, d_{32}, of the emulsion globule should be determined respectively.

1. Distribution Coefficient, α':

   Because the membrane phase contains emulsifier, violent vibrations should be avoided while measuring α'. In this case, both the aqueous and the membrane phases were mixed at 100 rpm until the equilibrium was reached. After settling, α' was calculated to be 0.266 by measuring the concentration of acetic acid in aqueous phase.

2. Effective Diffusivity, D_e:

   The D_e of acetic acid in the saturated zone of the emulsion globule was estimated from the Jefferson-Witzell-Sibbett equation (11).

   The viscosity of the membrane phase was 3.53×10^{-3} Ns/m^2 (298 K) and the molar volume of acetic acid at its normal boiling point was 64.1×10^{-3} m^3/kmol. Therefore, at 298 K, $D_e = 5.43 \times 10^{-10}$ m^2/s.

3. Sauter Diameter, d_{32}, of the Emulsion Globule:

   Adpoting the photographic technique and using the equation $d_{32} = \frac{\Sigma n_i d_i^3}{\Sigma n_i d_i^2}$, the d_{32} at 400 and 500 rpm was found to be 1.08 and 0.90 mm, respectively.

4. Mass Transfer Coefficient, K, in External Boundary Layer:

   Skelland et al (14) suggested a correlative equation of a continuous phase under the conditions of lower interfacial tension as well as the liquid depth being equal to the diameter of stirrer, after studying the value of K of carboxylic acid in the aqueous phase.

$$\frac{K}{\sqrt{nD_w}} = 1.864 \times 10^{-6} \phi^{-0.287} \left(\frac{D}{T}\right)^{0.548} N_{Re}^{1.371} N_{We}^{-0.095} \qquad (29)$$

K is calculated to be 1.38×10^{-5} and 2.01×10^{-5} m/s at 400 and 500 rpm, respectively. The value of M is, therefore, 0.0730 and 0.0601, respectively.

Making a comparison between model predictions and experimental data in Fig. 5, it is apparent that when M equals zero, that is to say, mass transfer resistance in the boundary layer of the external phase is neglected and the predictions are calculated from the Advancing Front Model, the deviation between the predictions and the experimental data is appreciably larger. When the mass transfer resistance in the boundary layer of the external phase is taken into account, the calculated values are in good agreement with the experimental data on batch extraction of acetic acid from wastewater.

THE SWELL OF EMULSION

Swell phenomenon implies the greater change of emulsion volume while the emulsion is being agitated in the external phase. The swell is harmful to the LSM in two aspects. Firstly, it dilutes the internal phase and makes the recycle of the emulsion difficult. Secondly, it changes the hydrodynamic properties of the emulsion so that the design of equipment becomes difficult. The swell is caused by two factors, one is entrainment during mixing, the other is water permeation from external to internal due to the osmotic pressure difference. In this paper, the factors affecting swell and the swelling mechanism of entrainment have been studied.

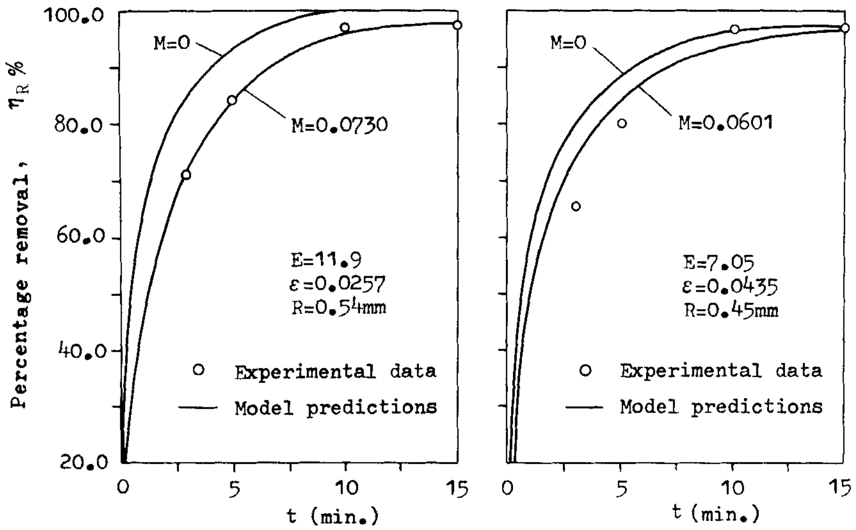


Fig. 5 Comparison of model predictions with experimental data for the removal of acetic acid

The Effect of Entrainment

Deionized water or equal concentration of NaCl solution was used as both internal and external phases. The results obtained are plotted in line b of Fig. 6. It is obvious that the entrainment swell is proportional to the mixing time, but even after stirring for 60 min, the percentage swell is still below 8%. This shows that the effect of entrainment on swell is fairly small compared to other effects described below.

The Effect of Osmotic Pressure Difference

The experiments were carried out under the following two conditions, 3M NaCl or 3M NaOH solution was used as internal

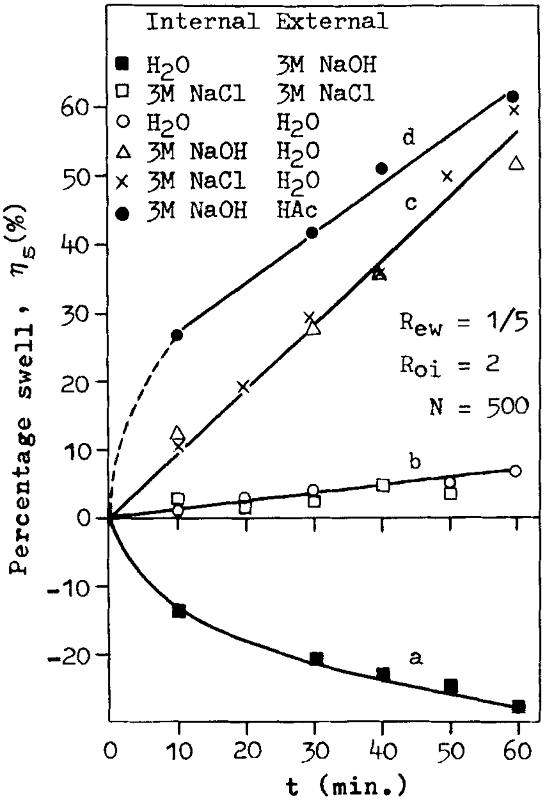


Fig. 6 Swell of emulsion at various conditions

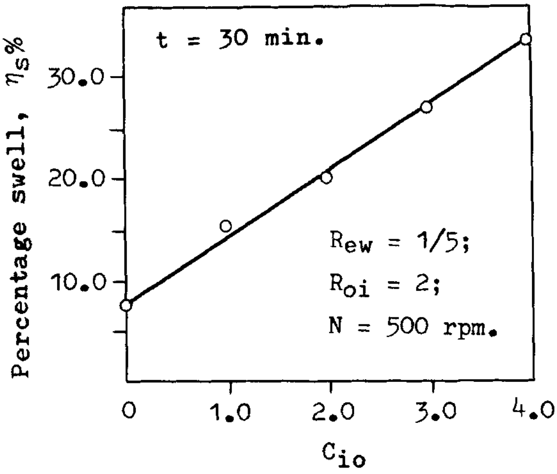
phase, while deionized water was used as the external phase in both cases. The data illustrated in line c of Fig. 6 represent the summation of the effects of entrainment and water permeation. It may be seen by comparing line b with line c that water permeation influences the swell more than entrainment does.

When the experimental condition is inversed, namely, deionized water as internal and 3M NaCl solution as external, water will be transferred from internal phase to external phase as shown in line a of Fig. 6.

Increasing the NaOH concentration in the internal phase results in increasing the osmotic pressure difference between the internal and external phase, as well as raising the swelling rates. It is found from Fig. 7 that the percentage swell varies with the NaOH concentration in the internal phase.

The Effect of Mass Transfer of Acetic Acid

By comparing curve d in Fig. 6, where 3M NaOH was used as internal and 0.0861M HAc as external, with curve c, where deionized water as external, it is evident that in the case of mass transfer of acetic acid, the swell of emulsion increases, especially at the beginning of stirring. The reason may be that



External phase: Deionized water

Fig. 7 The effect of internal NaOH concentration on swell the higher concentration of acetic acid in the external phase results in a higher equilibrium concentration in the membrane phase, and an increment of polar matter in the membrane results in an increase in the solubility of water, so the transfer rate of water towards the internal phase becomes more rapid.

The Effect of Surfactant and Carrier Additives

Table 1 shows that the higher the concentration of surfactant, the more the swell of emulsion. Because the solubilization of surfactant results in increasing the solubility of water in the membrane phase, the transfer of water can be accelerated, thus the swell increases. The addition of carrier TBP in the membrane also promotes the swell as shown in Table 1.

The Effect of Redispersion and Stirring Speed

When stirring speed reached 3, 5 and 10 minutes, stirring paused for one minute so that the emulsion and the external phase could be separated and then stirred again. It was found from Table 1 that, after coalescence and redispersion took place three times, the swell of the emulsion increased and the external phase water disappeared while mass transfer of acetic acid occurred. It, therefore, seems that a multistage mixer-settler is unsuitable for liquid membrane operation.

Fig. 8 shows that the swell rapidly increases with stirring speed. These results can be explained as follows: because an excess of surfactant exists in the membrane, an adsorbing layer of surfactant is formed on the curved surface where the membrane phase contacts entrained water droplets to reduce the interfacial tension. This procedure is shown graphically in Fig. 9.

TABLE 1
The Effect of Surfactant and Carrier on the Emulsion Swell

C_s (%)	Internal phase	External phase	s(%)	Notes
3.0	3.03M NaOH	H_2O	18.5	
5.0	3.03M NaOH	H_2O	23.0	
7.0	3.03M NaOH	H_2O	26.6	
8.7	3.03M NaOH	H_2O	27.4	
8.7	3.03M NaOH	H_2O	40.7	Coalescence and redispersion three times
8.7	3.00M NaCl	H_2O	29.6	
8.7	3.03M NaOH	0.0861M HAc	42.2	
8.7	3.03M NaOH	0.0861M HAc	46.7	1% TBP in membrane

$R_{ew} = 1/3$, $R_{oi} = 2$, $N = 500$ rpm, $t = 30$ min.

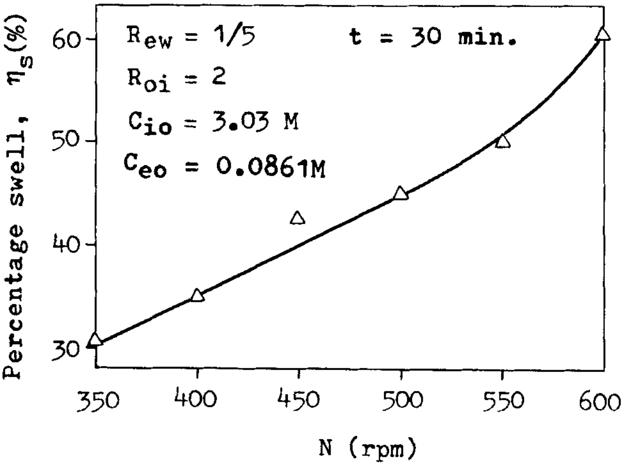


Fig. 8 The effect of stirring speed on the emulsion swell

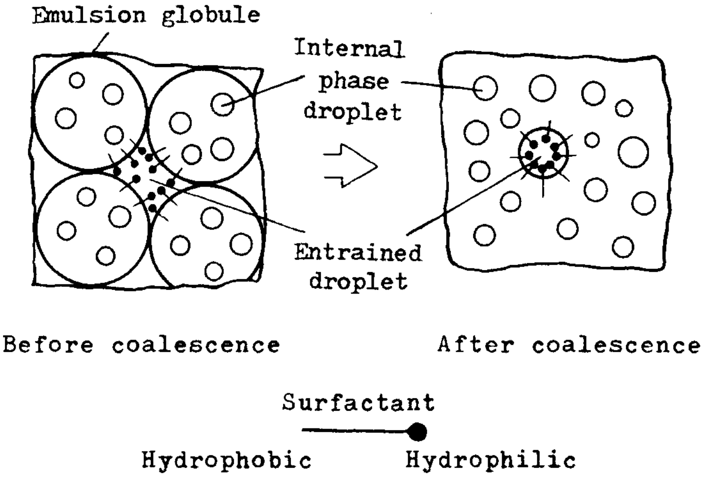


Fig. 9 Swelling mechanism of entrainment

According to Stokes' law, the subsidency velocity of rigid droplets in viscous fluid is proportional to the square of the drop diameter. The bigger entrained droplets are enough to settle from the emulsion by gravity force, but the smaller droplets are still left-in-place so that the volume of the emulsion expands. The more the repetition of coalescence and redispersion, the more the entrained droplets and swell.

In an agitated vessel, the emulsion globules are broken near the tip of the impeller blades and are swept away rapidly to parts of the vessel away from the impeller where they tend to coalesce. Hence, the emulsion globule size distribution at a given condition is caused by the dynamic balance of coalescence-redispersion (15), and the frequencies of coalescence-redispersion would be higher and the globules would be smaller when the stirring speed is faster (16). As the globule size decreases, the entrained droplets become too small to be settled from the emulsion, then the swell of emulsion rapidly increases.

CONCLUSIONS

A new diffusion controlled mass transfer model in the LSM has been developed. Resistance in both the external boundary layer and the membrane are considered simultaneously. Approximate analytical solutions of non-linear equations have been obtained by the perturbation method. The parameter M, included in the solutions, represents the ratio of resistance in external boundary layer to maximum resistance in emulsion globules. If M equals zero, the solutions are the same as those of the Advancing Front Model. Experimental data on batch extraction of acetic acid are in good agreement with the model predictions. Usually, the zero-order, or pseudo-steady state solution alone can be applied to describe the mass transfer process in the liquid membrane.

The swell of emulsion can be classified into two types - permeation and entrainment. Increasing the concentration of internal reagent and surfactant results in the increase of swelling rate. When the concentration of the internal reagent is higher, the effect of water permeation is more than that of entrainment on swell.

Repeated coalescence and redispersion, as well as vigorous stirring, will raise the swell due to entrainment. So it seems that the multistage mixer-settler is unsuitable for liquid membrane operation.

NOTATION

$B = (1-3/E)^{1/3}$

C - HAc concentration in saturated zone of emulsion globules, mol/l

C_{io} - initial NaOH concentration in internal phase, mol/l

C_e - HAc concentration in external phase, mol/l

C_{eo} - initial HAc concentration in external phase, mol/l

C_s - surfactant concentration in membrane (wt.%)

d_{32} - Sauter diameter of emulsion globules, m

D - impeller diameter, m

D_e - effective diffusivity in saturated zone of emulsion globules, m^2/s

D_w - diffusivity in external phase, m^2/s

$E = 3\frac{C_{io}V_{io}}{C_{eo}V_{eo}}$

$g = \frac{C}{\alpha C_{eo}}$

$h = \frac{C_e}{C_{eo}}$

h_o, h_1 - zero and first-order terms in perturbation expansion for h

$h^{(1)} = h_o + \varepsilon h_1$, first-order perturbation solution for h

K = mass transfer coefficient of acetic acid in external boundary layer, m/s

$L = \frac{1}{\alpha M}$

$M = \frac{D_e}{KR}$

n - stirring speed, rps

N - stirring speed, rpm

$N_{Re} = nD^2\rho_c/\mu_c$, agitation Reynolds number

$N_{We} = n^2D^3\rho_c/\sigma$, Weber number

r - radial coordinate in emulsion globules, m

R - radius of emulsion globules, m

R_{ew} - volume ratio of emulsion phase to external phase

R_f - reaction front position, m

R_{oi} - volume ratio of membrane phase to internal phase

t - time, min

T - diameter of agitation vessel, m

V'_e - volume of emulsion phase, l

V_i, V_m, V_e - volume of internal, membrane and external phase, respectively, l

$x = \frac{R_f}{R}$

Greek Letters

α = $(V_i+\alpha' V_m)/(V_i+V_m)$, distribution coefficient for acetic acid between external phase and saturated zone of emulsion at equilibrium

α' = $\frac{C_m}{C_e}$, distribution coefficient for acetic acid between membrane phase and external phase

ε - perturbation parameter, eq. 9

η = $\frac{r}{R}$

η_R = $(1 - \frac{C_e}{C_{eo}})\%$, percentage removal

η_s = $\frac{V'_e - V'_{eo}}{V'_{eo}}\%$, percentage swell

μ_c - viscosity of external phase, Ns/m^2

τ = $\frac{\varepsilon D_e t}{R^2}$

ϕ = $\frac{V_i+V_m}{V_i+V_m+V_e}$, volume fraction of emulsion

ρ_c - density of external phase, kg/m^3

σ - interfacial tension, N/m

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