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EFFECT OF EMULSION BREAKAGE ON SELECTIVITY IN THE SEPARATION OF HYDROCARBON MIXTURES USING AQUEOUS SURFACTANT MEMBRANES

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Summary

In liquid membrane separation processes emulsion breakage results in non-selective physical mixing of the feed mixture with the receiving solvent phase. In this paper a model is developed for describing the interphase transfer process, which takes emulsion breakage into account. The overall transfer is envisaged as a result of two parallel transfer mechanisms: (i) diffusive transport across the membrane and (ii) non-selective physical mixing of the feed with the receiving phase due to emulsion breakage. For selective removal of aromatics from non-aromatics in a feed mixture the "ideal" selectivity, β, obtained in the absence of non-selective breakage, will be given as the ratio of the products of the distribution coefficients times the diffusivity in the aqueous membrane phase. Experiments were carried out in a batch stirred cell to determine the permeation rates for a benzene–n-heptane mixture. From the experimentally observed selectivities the contribution due to emulsion breakage was estimated. This fractional breakage was in good agreement with values determined independently using a water-insoluble dye tracer technique, lending support to the developed model. Further experiments were carried out with the system 1-methylnaphthalene–dodecane, and breakage-corrected transfer rates were determined. The model developed in this paper, together with the experimental studies, sheds light on the mechanism of liquid membrane permeation and should aid in scaling-up processes for dearomatization of naphtha and kerosine.

1. Introduction

The removal of aromatic hydrocarbons from petroleum refinery streams such as naphtha and kerosene presents a difficult separation problem. The petroleum industry uses traditional separation processes such as liquid–liquid extraction, which are energy intensive, to effect these separations. An attractive possibility of a low energy alternative separation process lies in the utilisation of thin aqueous membranes stabilised by emulsification. This the Liquid Membrane Permeation (LMP) technique, a novel separation process shown to have

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potential applications in several diverse areas such as waste water treatment [1], hydrocarbon separations [2], and medical technology [3]. An industrial-scale plant using LMP for recovery of zinc was recently commissioned at Lenzing, Austria [4].

Aqueous membranes for hydrocarbon separations are generated by dispersing an o/w emulsion of the hydrocarbon mixture to be separated into a non-aqueous receiving "solvent" phase. The membrane interposes a barrier between the hydrocarbon feed phase and the receiving phase and allows selective transport of aromatics from the feed to receiving phase, primarily due to what is believed to be the much higher solubility of aromatics in the aqueous phase compared with non-aromatics [2, 5, 6]. Ideally, the membrane should be stable and prevent the feed from physically mixing with the "solvent" phase because such mixing would entail a loss in selectivity. Under these conditions, selectivity should be governed solely by the relative rates of transfer of compound types across the membrane phase; these relative rates are determined by the ratio of the products of distribution coefficients and diffusivity of transferring species in the aqueous phase. However, such ideal conditions do not always prevail and in practice what happens is that emulsion breakage can cause membrane rupture so that there is non-selective transport of a portion of the feed mixture into the solvent, resulting in loss in selectivity [7]. While there have been several investigations of the effect of various parameters on selectivity in a liquid membrane hydrocarbon separation [8–10], what has not generally been realised is that the "overall" mass transfer coefficients obtained in these permeation experiments reflect not only selective diffusive transport across the membrane but also non-selective mixing of feed with solvent. Mass transfer coefficients should therefore be corrected for breakage of emulsions in order for meaningful conclusions to be drawn.

In this paper we develop a model for the liquid membrane separation process taking proper account of emulsion breakage. An estimate of the extent of non-selective transport due to this breakage was made by application of the model equations to experimental data from liquid membrane separation of benzene–heptane mixtures; it was verified by independent measurements using a water-insoluble dye "tracer". This validated the modelling of the process, and the model equations were then used to interpret the experimental mass transfer data from liquid membrane separations of benzene–n-heptane and 1-methylnaphthalene–dodecane mixtures under conditions of varying permeation time, surfactant concentration and feed hydrocarbon/water ratio.

Model development

Emulsion breakage results in the entire contents of the feed droplet mixing with the "solvent" and can be considered as a "mixing" transport which does not occur through the membrane. We model the overall permeation process as a parallel-step process:

(1) A diffusional transmembrane transport which is selective.

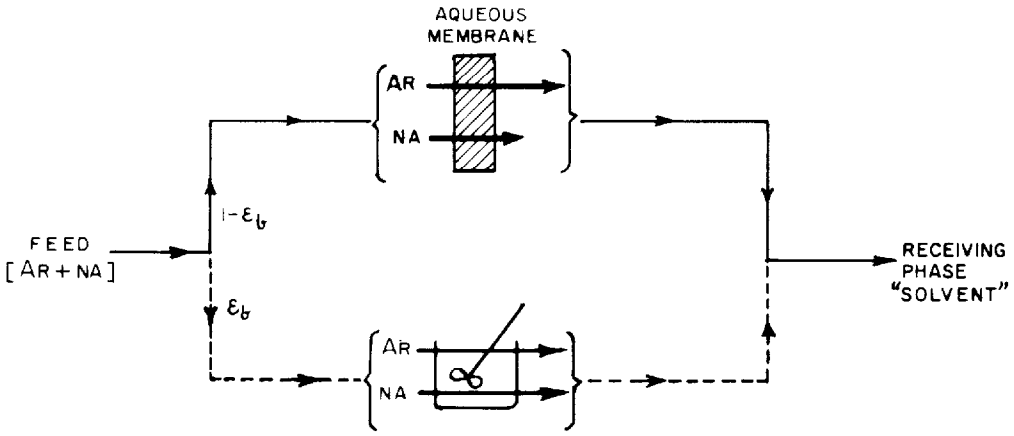

Fig. 1. Schematic diagram of parallel transport mechanism for liquid membranes.

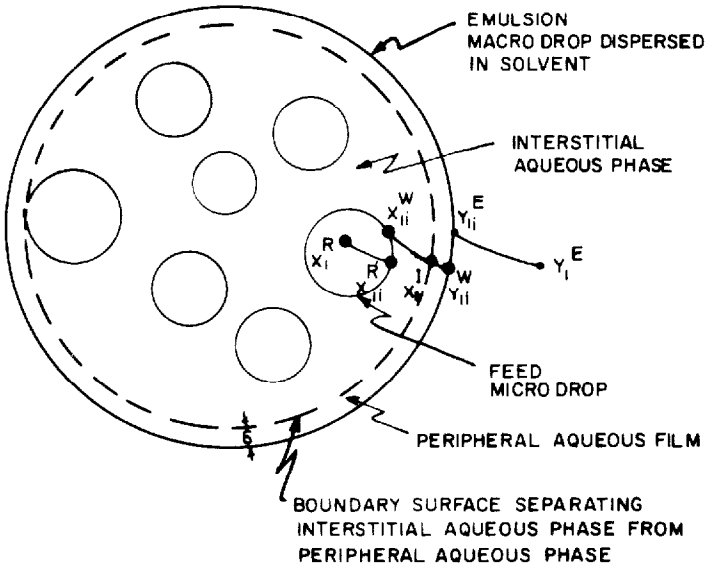

Fig. 2. Concentration profiles at time t for transmembrane transport.

(2) A non-selective "mixing" transport due to emulsion breakage.

A schematic diagram of the model is given in Fig. 1, where the feed mixture is considered to be made up of two compound types: aromatics (AR) and non-aromatics (NA). The fraction of the feed mixture which gets transferred to the solvent phase due to emulsion breakage is denoted by ϵ_b.

We first consider the diffusive transmembrane transfer. At time t, the concentration profile of the transferring aromatic species AR is shown schemati-

cally in Fig. 2. We note here that mass transfer is occurring through two interfaces of widely different areas. One of these, $a^{(1)}$, refers to that at the feed microdrop–water interface and the other, $a^{(2)}$, refers to that at the emulsion macrodrop–solvent interface. We have considered the aqueous membrane phase to consist of two regions, an interstitial aqueous phase between the feed microdrops and a peripheral aqueous film separated by a boundary surface, as shown in Fig. 2. Four sequential steps in the mass transfer process may be distinguished:

(i) Transfer within a microdrop of the emulsion; the mass transfer coefficient is denoted by k_1^{R}.

(ii) Transfer from the surface of a microdrop to the bulk interstitial aqueous phases; transfer coefficient denoted by $k_1^{\mathrm{W}(1)}$.

(iii) Transfer from the bulk interstitial aqueous phase to the surface of a macrodrop; transfer coefficient denoted by $k_1^{\mathrm{W}(2)}$. This transfer process is presumed to be governed by molecular diffusion across a peripheral film.

(iv) Transfer from the surface of a macrodrop to the bulk solvent phase; transfer coefficient k_1^{E}.

If we define an overall mass transfer coefficient, K_1^{E}, for the transfer process from feed to solvent, at steady state we have

$$N_1^{(1)}\, a^{(1)} = N_1^{(2)}\, a^{(2)} = K_1^{\mathrm{E}}\, a^{(2)}\, \rho^{\mathrm{E}}\, (x_1 - y_1) \tag{1}$$

where x_1 and y_1 refer to mass fractions of aromatics in the raffinate and extract phases, respectively. We can derive the following expression for the overall transfer coefficient, K_1^{E}:

$$\frac{1}{K_1^{\mathrm{E}} a^{(2)} \rho^{\mathrm{E}}} = \frac{1}{k_1^{\mathrm{R}} a^{(1)} \rho^{\mathrm{R}}} + \frac{1}{M_1 k_1^{\mathrm{W}(1)} a^{(1)} \rho^{\mathrm{W}}} + \frac{1}{M_1 k_1^{\mathrm{W}(2)} a^{(2)} \rho^{\mathrm{W}}} + \frac{1}{k_1^{\mathrm{E}} a^{(2)} \rho^{\mathrm{E}}} \tag{2}$$

The small sizes of the feed microdrops (usually around 1–10 μm) and emulsion macrodrops (of diameter around 1–3 mm), coupled with the fact that surfactants are present, would suggest that rigid drop behaviour may be assumed for both microdrops and macrodrops. For transfer within and outside rigid spheres, the Newman model [11] gives, for sufficiently high Fourier numbers, Sherwood numbers of 6.58 and 2, respectively. This, along with the diffusivity data, allows an estimation of the coefficients k_1^{R}, $k_1^{\mathrm{W}(1)}$ and k_1^{E}. Estimation of $k_1^{\mathrm{W}(2)}$ requires an estimate of the effective thickness δ of the peripheral film; this is possible assuming δ to be given by the intermicrodrop distance, which can be calculated from a knowledge of microdrop and macrodrop diameters. The transfer coefficient $k_1^{\mathrm{W}(2)}$ then is

$$k_1^{\mathrm{W}(2)} = D_1^{\mathrm{W}}/\delta \tag{3}$$

The differential material balance for component 1 in the extract phase is

$$\frac{dE_1}{d\xi}=\frac{d(Ey_1)}{d\xi}=N_1^{(2)}a^{(2)}V^E\tau \tag{4}$$

Noting that the flux N_1 can be written as

$$N_1^{(2)}=\rho^E K_1^E(x_1-y_1)+y_1(N_1^{(2)}+N_2^{(2)}) \tag{5}$$

we get from eqns. (4) and (5)

$$E\frac{dy_1}{d\xi}+y_1\frac{dE}{d\xi}=\rho^E K_1^E(x_1-y_1)a^{(2)}V^E\tau+y_1(N_1^{(2)}+N_2^{(2)})a^{(2)}V^E\tau \tag{6}$$

An overall balance gives

$$\frac{dE}{d\xi}=(N_1^{(2)}+N_2^{(2)})a^{(2)}V^E\tau \tag{7}$$

so that combining eqns. (6) and (7)

$$\frac{dy_1}{d\xi}=\frac{\rho^E K_1^E a^{(2)}V^E\tau}{E}(x_1-y_1) \tag{8}$$

We next consider the "mixing" transfer due to emulsion breakage that occurs parallel to transmembrane diffusion. We have the following relations at any instant

$$\frac{dE}{d\xi}=-\frac{dR}{d\xi};\qquad \frac{d(Ey_1)}{d\xi}=-\frac{d(Rx_1)}{d\xi} \tag{9}$$

Now as the "mixing" transfer is non-selective, the raffinate phase composition will not change during this process, so that $dx_1/d\xi=0$. Thus we get from eqn. (9) the change in the extract phase composition due to emulsion breakage as

$$E\frac{dy_1}{d\xi}=-(x_1-y_1)\frac{dR}{d\xi} \tag{10}$$

Referring to Fig. 1, we may write the net composition change of the extract phase due to both diffusive and mixing transfer process as

$$\frac{dy_1}{d\xi}=-\epsilon_b\frac{(x_1-y_1)}{E}\frac{dR}{d\xi}+\frac{(1-\epsilon_b)\rho^E K_1^E a^{(2)}V^E\tau(x_1-y_1)}{E} \tag{11}$$

Both amounts of raffinate and extract phase, R and E, respectively, vary with time. Let R^0 and E^0 represent the initial amounts. We then have the material balance relationships

$$E=E^0+R^0-R;\qquad x_1=(R^0x_1^0-y_1E)/R \tag{12}$$

Also, we define the number of overall transfer units NTU_1 as

$$\mathrm{NTU}_1 = \frac{\rho^{\mathrm{E}} K_1^{\mathrm{E}} a^{(2)} V^{\mathrm{E}} \tau}{E^0} \tag{13}$$

The variation of R with time can be expressed empirically as

$$R = R^0 f(\xi) \tag{14}$$

Using the relationships given in eqns. (12) and (13) we can integrate eqn. (11) from $\xi = 0$ to $\xi = 1$ to give

$$\mathrm{NTU}_1 = \frac{\dfrac{1}{1+m} \ln\left(\dfrac{mx_1^0}{mx_1^0 - y_1(1+m)}\right) + \epsilon_b \displaystyle\int_0^1 \frac{f'(\xi)\,\mathrm{d}\xi}{[1+m-mf(\xi)]f(\xi)}}{\dfrac{(1-\epsilon_n)}{m} \displaystyle\int_0^1 \frac{\mathrm{d}\xi}{f(\xi)\,[1+m-mf(\xi)]}} \tag{15}$$

where $m = R^0/E^0$ represents the initial feed/solvent ratio and x_1^0 is the initial mass fraction of benzene (component 1) in the feed. If we denote β as the selectivity, defined as the ratio of the numbers of transfer units from species 1 and 2, i.e., $\beta = \mathrm{NTU}_1/\mathrm{NTU}_2$, then the fractional breakage can be calculated from

$$\epsilon_b = \frac{\dfrac{\beta}{1+m} \ln\left(\dfrac{mx_2^0}{mx_2^0 - y_2(1+m)}\right) - \dfrac{1}{1+m} \ln\left(\dfrac{mx_1^0}{mx_1^0 - y_1(1+m)}\right)}{(1-\beta) \displaystyle\int_0^1 \frac{f'(\xi)\,\mathrm{d}\xi}{f(\xi)[1+m-mf(\xi)]}} \tag{16}$$

Equation (16) may be used to estimate the fractional breakage from liquid membrane permeation experiments if we assume that the selectivity β is equal to the ratio of the product of the distribution coefficient times the diffusivity of the species 1 (aromatics) and 2 (non-aromatics) in the aqueous membrane phase, i.e., $\beta = K_1 D_1 / K_2 D_2$. This simplification follows from the fact that the mass transfer process is controlled by the third term on the right-hand side of eqn. (2). This can be verified by substituting the values of the various parameters as listed in Table 1.

Experimental

The liquid membrane permeation experiments were carried out at 30°C in a thermostatted glass mixer–settler unit of 300 ml capacity as shown in Fig. 3. The model hydrocarbon mixtures, along with the respective surfactants and "solvents" used, are listed in Table 2, as is the range of parameters studied. The oil-in-water emulsions were prepared by vigorous agitation of the feed with aqueous surfactant solution at 4000 rpm for 15 minutes and then mixed

TABLE 1

Hydrodynamic, thermodynamic and physical property values used in the calculations quoted in the text

Property	Value used	Source
Dispersed phase microdrop diameter	8 μm	own measurements[a]
Emulsion macrodrop diameter	2 mm	own measurements[a]
Volume fraction dispersed phase in emulsion	0.5	own data
Volume fraction emulsion in solvent	0.5	own data
Density of extract phase	820 kg-m^{-3}	experimental
Density of raffinate phase	790 kg-m^{-3}	experimental
Distribution coefficient of benzene between aqueous and hydrocarbon phases at 30°C	1.78×10^{-3}	Ref. [12]
Distribution coefficient of n-heptane between aqueous and hydrocarbon phases at 30°C	2.94×10^{-6}	Ref. [12]
Diffusivity of benzene in hydrocarbon phase	3.42×10^{-9} m^2-sec^{-1}	Ref. [13]
Diffusity of n-heptane in waer	0.91×10^{-9} m^2-sec^{-1}	Ref. [13]
Diffusivity of benzene in water	1.32×10^{-9} m^2-sec^{-1}	Ref. [13]
Volume of extract phase	6.25×10^{-5} m^3	experimental value
Thickness of peripheral aqueous film	1 μm	estimated

[a]Drop size measurements were made by photomicrography using a Leitz microscope.

with "solvent" at 650 rpm in the same unit. The phases were then settled and analysed by an azeotropic distillation procedure standardized and reported elsewhere [14]. In some of the experiments with benzene–n-heptane feed mixtures, 1-pentanol was added to the kerosene "solvent" to aid phase separation at the end of the permeation experiment. This additive was, however, not effective in the 1-methylnaphthalene–dodecane system; in this case centrifugation at 800–1000 rpm was necessary.

In addition to these mass transfer experiments, the fractional breakage was measured independently using a water-insoluble dye tracer technique [2]. A known quantity of dye was added to the hydrocarbon feed mixture before emulsification. As the dye molecules are insoluble in the membrane phase, there will be no diffusional transmembrane transport of these molecules into the solvent phase but there will be only "mixing" transport due to emulsion breakage. From the concentration of the dye in the solvent phase after each permeation experiment, the fractional breakage of the emulsion can be calculated. Dye concentrations in the solvent were determined from absorbance measurements at 480 nm using a Shimadzu UV-240 spectrophotometer.

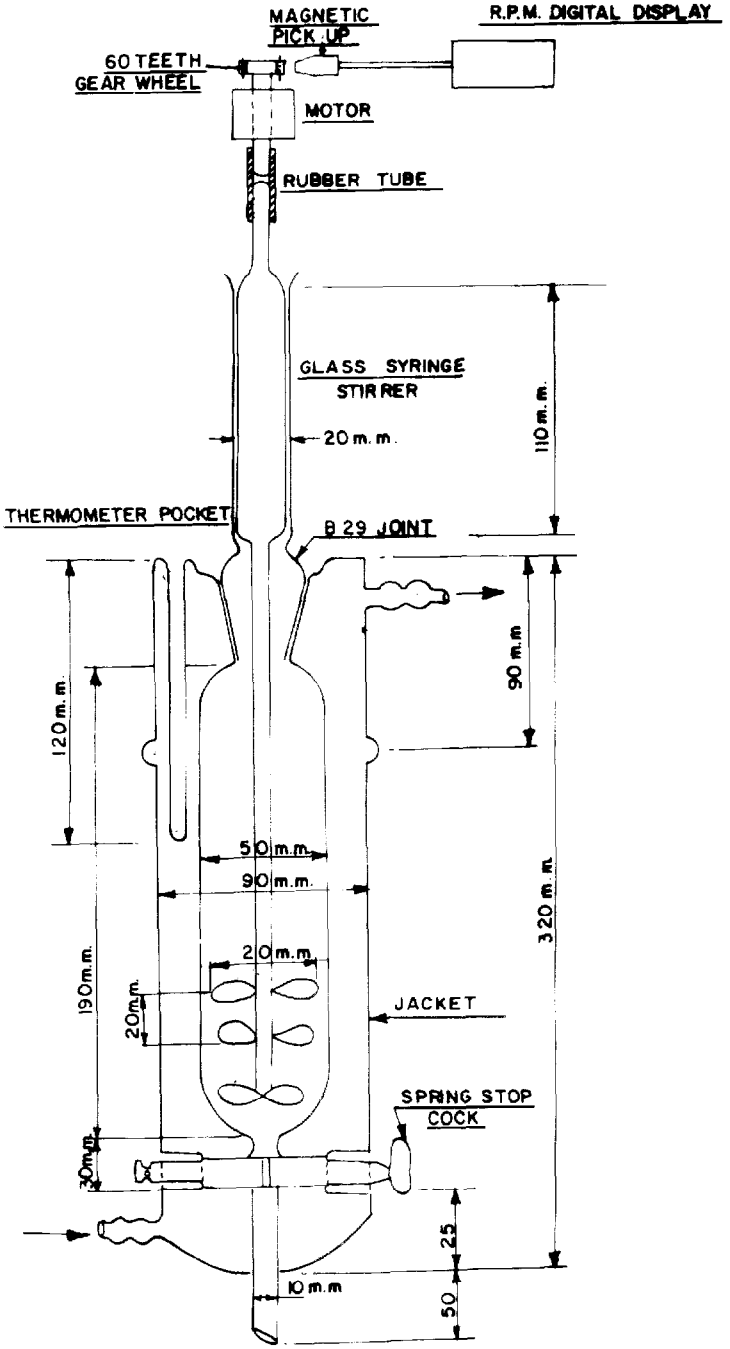

Fig. 3. Mixer-settler unit used in liquid membrane permeation experiments.

TABLE 2

Liquid membrane systems and parameters studied

Model hydrocarbon feed	Surfactant	Solvent	Parameters studied
Benzene–n-heptane [50:50] (w/w)	Hyoxd X200	kerosene	(1) permeation time
Benzene–n-heptane [50:50] (w/w)	Hyoxd X200	kerosene–1-pentanol [80:20] (w/w)	(1) permeation time (2) surfactant concentration (3) hydrocarbon feed/water ratio
1-Methyl-naphthalene–dodecane [27:73] (w/w)	Noigen DK-30	n-heptane	(1) permeation time (2) surfactant concentration

Results and discussion

Table 3 gives the set of experimentally measured compositions of benzene (1) and n-heptane (2) in the extract phase under a varying set of conditions. Using the diffusivity and solubility data of benzene and n-heptane in water as given in Table 1, the "ideal" selectivity, to be expected in the absence of non-selective breakage β, was calculated to be 878. The function $f(\xi)$ in eqn. (14) was evaluated from a fit of actual measured data and found to be well represented by

$$f(\xi) = \frac{1 + \dfrac{0.0737\xi}{R^0}}{(1 + 6.29\xi)} \tag{17}$$

The fractional breakage, ϵ_b, and the overall mass transfer coefficient, $K_1^E a^{(2)}$, were calculated from measured data using eqns. (15)–(17); they are also reported in Table 3. The values of ϵ_b calculated in this manner are compared in Fig. 4 with direct measurements of this parameter using the water-insoluble dye tracer technique. The excellent agreement beween directly measured fractional breakage and the values estimated using eqn. (16) provides a strong proof of the validity of the model developed in this paper. Using the values of the various parameters listed in Table 1, the estimate of the overall mass transfer coefficient $K_1^E a^{(2)}$ using eqn. (2) is 0.002 sec^{-1} for the benzene–n-heptane system, which is in the range of the experimental determined *breakage corrected* mass transfer coefficients, see Table 3. This provides further indirect validation of the model and assumptions made in this paper.

In Table 4 we report the experimentally measured compositions of 1-methylnaphthalene (1) and dodecane (2) in the extract phase, and fractional

TABLE 3

Experimental data on separation obtained in batch mixer-settler unit for the system benzene-n-heptane

Parameter varied	m	R^0 (kg)	y_1	y_2	ϵ_b	$K_1^E a^{(2)}$ (sec^{-1})	Solvent used
Permeation time, (sec)							
120	0.368	0.0265	0.1152	0.0086	0.078	0.00324	kerosine
180	0.353	0.0251	0.1095	0.0082	0.082	0.00202	
240	0.400	0.0247	0.1276	0.0100	0.090	0.00210	
300	0.380	0.0250	0.1100	0.0120	0.115	0.00120	
Permeation time, (sec)							
60	0.484	0.0242	0.1072	0.0170	0.14	0.00493	kerosine-
120	0.495	0.0247	0.1345	0.0200	0.16	0.00403	1-pentanol
180	0.516	0.0258	0.1421	0.0245	0.18	0.00299	[80:20]
240	0.520	0.0260	0.1472	0.0229	0.16	0.00242	(w/w)
300	0.500	0.0250	0.1423	0.0312	0.25	0.00206	
Surfactant concentration (wt.%)							
0.10	0.493	0.0246	0.1346	0.0226	0.18	0.00277	kerosine-
0.20	0.480	0.0240	0.1345	0.0253	0.21	0.00299	1-pentanol
0.30	0.510	0.0253	0.1377	0.0226	0.17	0.00277	[80:20]
0.40	0.500	0.0251	0.1388	0.0224	0.17	0.00291	(w/w)
0.50	0.497	0.0249	0.1377	0.0208	0.18	0.00284	
Hydrocarbon feed/water							
1.00	0.510	0.0253	0.1377	0.0226	0.17	0.00277	kerosine-
1.25	0.550	0.0274	0.1499	0.0227	0.15	0.00309	1-pentanol
1.50	0.612	0.0306	0.1571	0.0295	0.16	0.00310	[80:20] (w/w)

Note: In all the above experiments, the fractional breakage ϵ_b was calculated from the theoretical model using $\beta = 878$, and the following parameters were kept constant: benzene mass fraction in feed, $x_1^0 = 0.5$; rpm during emulsification = 4000; and rpm during permeation = 650.

breakage under a varying set of conditions. The form of the function $f(\xi)$ for this system was evaluated from measured data to be

$$f(\xi) = 1 - 0.125\xi \tag{18}$$

The experimental overall volumetric mass transfer coefficients $K_1^E a^{(2)}$ calculated from measured data using eqns. (15) and (16) are also given in Table 4.

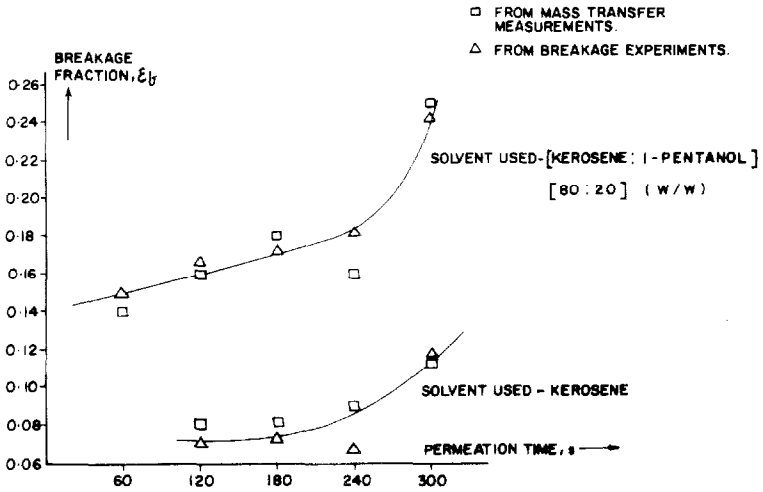

Fig. 4. Fractional breakage from direct measurements and from calculations for the system benzene–n-heptane.

TABLE 4

Experimental data on separation obtained in batch mixer-settler unit for the system 1-methylnaphthalene–dodecane

Parameter varied	m	R^0 (kg)	y_1	y_2	ϵ_b	$K_1^E a^{(2)}$ (sec^{-1})	Solvent used
Permeation time (sec)							
180	0.501	0.0201	0.0305	0.006-	0.023	0.00075	n-heptane
240	0.510	0.0202	0.361	0.0081	0.045	0.00070	
300	0.503	0.0201	0.0412	0.0060	0.044	0.00067	
Surfactant concentration (wt.%)							
0.2	0.500	0.0201	0.0301	0.0150	0.074	0.00046	n-heptane
0.3	0.511	0.0205	0.0303	0.0080	0.045	0.00045	
0.4	0.502	0.0199	0.0350	0.0060	0.054	0.00056	
0.5	0.500	0.0201	0.0291	0.0081	0.054	0.00043	

Note: In all the above experiments, fractional breakage ϵ_b was measured by independent dye tracer experiments, and the following parameters were kept constant: 1-methylnaphthalene mass fraction in feed, $x_1^0 = 0.27$; rpm during emulsification = 4000; rpm during permeation = 650.

From the data reported in Tables 3 and 4, it is seen that fractional breakage, ϵ_b, is significantly higher for benzene–n-heptane systems where 1-pentanol is present in the "solvent" phase than for both 1-pentanol-free or 1-methylnaphthalene-dodecane systems. This could be due to the high spreading coefficient of 1-pentanol at oil/water interfaces, which can cause emulsion breakage.

For both systems, fractional breakage is seen to increase with permeation time, suggesting that emulsion stability decreases with increasing contact time. With increased contact time, $K_1^E a^{(2)}$ remains fairly constant for 1-methylnaphthalene–dodecane systems but decreases for benzene–n-heptane systems. This suggests that coalescence effects leading to a decrease in interfacial area are more predominant with benzene–n-heptane systems. The fractional breakage and overall volumetric mass transfer coefficients are fairly insensitive to changing surfactant concentration for both systems. This suggests that, over the range studied, this parameter does not affect the system mass transfer behaviour. With increasing feed hydrocarbon/water ratio in the emulsion it is expected that the thickness of the aqueous membrane decreases. Therefore $K_1^E a^{(2)}$ should increase, as is seen to be the case from the data reported in Table 3.

Conclusion

A mathematical model has been developed which considers the overall process of hydrocarbon permeation through liquid membranes as a combination of two processes in parallel, a non-selective transfer due to emulsion breakage and a selective diffusional transfer through the membrane. The model has been verified by comparing the fractional breakage predicted from mass transfer measurements with direct measurements of this parameter. The model has been used to interpret mass transfer data from liquid membrane experiments with two model hydrocarbon feed mixtures under varying conditions. The model provides a better insight into the mechanism of mass transfer in liquid membranes. The estimates of the mass transfer coefficients which have been corrected for emulsion breakage, provided by the model, will be useful in scale-up of the process for dearomatisation of naphtha and kerosine.

List of symbols

a	interfacial area per unit volume of extract phase, m^{-1}
D	molecular diffusivity, m^2-sec^{-1}
d	diameter, m
E	mass of extract phase, kg
K	distribution coefficient between aqueous and organic phases
K	overall mass transfer coefficient, m-sec^{-1}
k	individual mass transfer coefficient, m-sec^{-1}

m initial feed/solvent ratio, –
N flux of component across interface, kg-m^{-2}-sec^{-1}
R mass of raffinate phase, kg
Sh Sherwood number, –
t time, sec
V volume, m^3
x mass fraction of component in raffinate phase
y mass fraction of component in extract phase

Greek symbols

β selectivity; $\beta = \mathrm{NTU}_1/\mathrm{NTU}_2$
δ effective film thickness, m
ρ mass density, kg-m^{-3}
ϵ_b fractional breakage
ξ dimensionless time, $\xi = t/\tau$
τ permeation time, sec

Subscripts

1,2 aromatic and non-aromatic compound, respectively
i interfacial value
I denotes boundary surface separating interstitial aqueous phase from peripheral film (see Fig. 2)

Superscript

E extract phase
R raffinate phase
W aqueous phase
(1), (2) denote interfaces at feed microdrop–water and emulsion macrodrop–solvent, respectively
0 initial value

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