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A SCALE-UP OF STIRRED TANK CONTACTORS FOR THE LIQUID MEMBRANE PERMEATION OF HYDROCARBONS

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ABSTRACT

The scale-up of stirred tank contactors on the drop size of emulsion-solvent dispersion and the mass transfer coefficient of hydrocarbon in oil-in-water O/W emulsions (dispersed phase) are discussed. Correlative equations to estimate the drop sizes and the mass transfer coefficient are proposed without regard to tank size and material systems. The consumption of power created by stirring one unit of liquid tank volume is an applicable scale-up standard for the drop size of O/W emulsion and the mass transfer coefficient of hydrocarbons in the emulsion.

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Key Words: Hydrocarbon separation; Liquid membrane permeation; Scale-up; Stirred tank; Mass transfer coefficient

INTRODUCTION

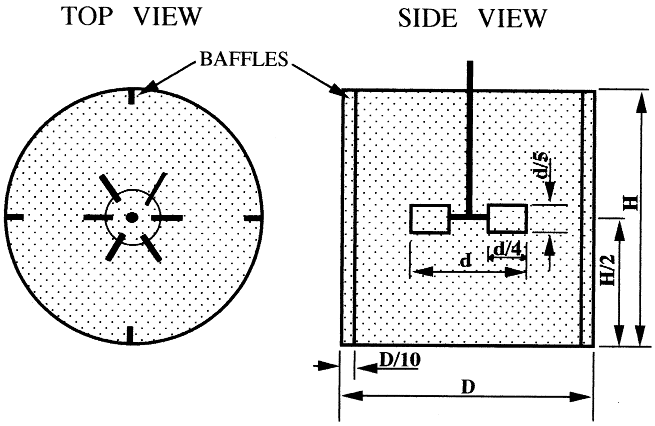
New separation technologies for hydrocarbon mixtures can be applied to separate the available aromatic hydrocarbons (especially 2,6-dimethylnaphthalene) in the light cycle oil (1) and the toxic aromatic hydrocarbons (especially benzene) in automobile gasoline (2). In the petroleum refinery industry, a new separation technique for hydrocarbon mixtures would be extensively studied and developed as an alternative to the conventional separation processes such as extraction, extractive distillation, and azeotropic distillation. Liquid membrane permeation (LMP), which was developed by N. N. Li (3–5), is known to be a promising separation technique because it separates hydrocarbon mixtures with relatively high selectivity and because it might be a less energy-consuming operation.

Although LMP has been known to be highly selective in hydrocarbon separation (3–10), industry has yet to utilize LMP. Therefore, this paper, as an examination of the industrial utilization of LMP, introduces a scale-up of the stirred tank that was used by several other researchers. In this work, liquid membrane separation of hydrocarbons was carried out to examine the scale-up of a stirred tank as a contactor of oil-in-water (O/W) emulsion and solvent t. Using batch stirred tanks of 2 sizes, we measured the sauter mean diameter of the O/W droplet dispersed in an oil-in-water-in-oil (O/W/O)-type emulsion, and then we conducted permeation experiments by using 2 different feed hydrocarbons (toluene + heptane and benzene + hexane) and 2 kinds of liquid membranes (saponin aqueous solution and sulfolane aqueous solution). Subsequently, mass transfer coefficients of the dispersed phase, using batch stirred tanks of 2 sizes, were measured. Also, the mass transfer coefficient for the dispersed phase for a batch operation was compared with that of a continuous operation.

EXPERIMENTAL

Apparatus and Method

The emulsification apparatus and methods for this procedure were the same as those described in our previous report (11). A schematic diagram and the dimensions of the 2 batch stirred tanks are presented in Fig. 1. Two glass tanks with 4 baffles each were used to compare their respective effects on per-



	D	d	d/D	H/D	V
Tank	[m]		[-]		[m3]
I	0.08	0.050	0.625	1	$4.0\text{x}10^{-4}$
II	0.15	0.094	0.625	1	$2.62\text{x}10^{-3}$

Figure 1. Experimental apparatus.

meation. A turbine-type impeller with the 6 flat blades was located at the center of the liquid.

Procedures for this permeation experiment were the same as those reported in our previous work (11). After a contact run of a specified period, the O/W/O emulsion was settled, and the volumes of the O/W emulsion phase and the solvent phase were measured. In all the runs, the solvent phase was analyzed by gas chromatography (Shimadzu Co, GC-8A) to determine the mass fraction of components in the outer oil phase. However, the O/W emulsion phases obtained in all the runs were demulsified by adding acetone. After adding the acetone, the composition of the oil phase was different from that of the O/W emulsion phase because part of the hydrocarbon in the O/W emulsion phase had shifted to the aqueous phase. Therefore, the oil to aqueous phase ratio was sufficiently increased by adding hydrocarbons that were not in the original system (feed, solvent); the content of the hydrocarbons in the aqueous phase should be ignored. This oil phase was separated and then washed by water to remove acetone. Then it was analyzed, and the composition of the inner phase was determined.

Methods for measuring the diameter of the O/W emulsion dispersed in the O/W/O emulsion, which was formed by the contact of an O/W emulsion and a solvent, were the same as those for our previous work (11).

System and Conditions

Material systems used and experimental conditions are presented in Table 1. Two mixtures of aromatic and nonaromatic compounds were used as the hydrocarbon feed: toluene (T) + heptane (Hp) (Feed I) and benzene (B) + hexane (Hx) (Feed II). The aqueous solution (Membrane I) contained saponin as an emulsifying agent, and the aqueous solution (Membrane II) contained sulfolane as a permeability promotion agent. These aqueous solutions were used as liquid membrane phases. Hexane and 2,2,4-trimethylpentane were used as solvents of Feed I and Feed II, respectively. In this work, hexane and 2,2,4-trimethylpentane were used as solvents because their solubilities in water are so low that their permeation through a liquid membrane is negligible. The emulsion made by Feed I and Membrane I was used to measure the diameter of the O/W emulsion dispersed in the O/W/O emulsion. Also, the emulsions made by (Feed I and Membrane I), (Feed II and Membrane I), and (Feed II and Membrane II) were used to measure the permeation rates of the hydrocarbon components in the O/W emulsion.

Stirring speeds were varied to measure a sauter mean diameter (d_{32}) for the O/W emulsion dispersed in the O/W/O emulsion, while the contact time of the O/W emulsion and the solvent was kept constant at 80 seconds. In this work, we assumed that the d_{32} measurement would be attained when the diameter of the O/W emulsion in the stirred tank had reached a steady state, irrespective of the stirring speeds. Meanwhile, stirring times for the permeation experiments were

Table 1. Systems and Experimental Conditions

Systems		
Feed	I	Toluene + Heptane, $\times_{T,0} = 0.5$
	II	Benzene + Hexane, $\times_{B,0} = 0.5$
Membrane	I	Water + Saponin (for Feed I, II) $C_S = 0.002$
	II	Water + Saponin + Sulfolane (for Feed II) $C_S = 0.002$, $C_{SUL} = 0.24$
Solvent	I	Hexane (for Feed I)
	II	2,2,4-Trimethylpentane (for Feed II)
Experimental conditions		
ϕ_d [–]	0.1	
$\phi_{o,0}$ [–]	0.5	
N [s^{-1}]	approximately 5.83–18.5	
T [s]	approximately 0–300	
T [°C]	30	

varied to discover the tendency of the time course curve for the permeation rates of hydrocarbon components at fixed stirring speed.

RESULTS AND DISCUSSION

Scale-Up on the Drop Size of the O/W Emulsion

In our previous work (11) using tank I, we reported a simplified empirical equation for d_{32}

$$d_{32} = \frac{\sum n_i \cdot d_i^3}{\sum n_i \cdot d_i^2}$$

of the O/W emulsion dispersed in the O/W/O emulsion.

$$\frac{d_{32}}{d} = 0.153 \cdot \mathrm{We}^{-0.6}$$
$$2 \times 10^2 < \mathrm{We}\left[= \left(\frac{N^2 \cdot d^3 \cdot \rho_d}{\sigma}\right)\right] < 5 \times 10^3 \tag{1}$$

The We number was determined through the use of the calculated value $[\rho_d = \rho_o \cdot \phi_o + \rho_W \cdot (1 - \phi_o)]$ of the density of the dispersed (O/W emulsion) phase (ρ_d) and the experimental value for the interfacial tension between the dispersed phase and the solvent (σ) of Egashira, Sugimoto, and Kawasaki (12), who obtained the value by using the same material systems we did in this work. The calculated value of ρ_d and the experimental value of σ were 887.5 kg/m^3 and 2.24×10^{-2} kg/s^2, respectively. In our previous work (11), we also reported Eq. (2) for the specific surface area $[a = (6 \cdot \phi_d)/d_{32}]$ between the dispersed phase and the solvent, and Eq. (3) for the thickness of a liquid membrane (δ) in the simplified O/W emulsion droplet derived from the result of Eq. (1).

$$a = \frac{(6 \cdot \phi_d)}{(0.153 \cdot d \cdot We - 0.6)} \tag{2}$$

$$\delta = 0.0765 \cdot d \cdot (1 - \phi_o^{1/3}) \cdot \mathrm{We}^{-0.6} \tag{3}$$

Equations (1, 2, and 3) can be expressed by using the impeller speed (N) and impeller diameter (d) as follows.

$$d_{32} \propto N^{-1.2} \cdot d^{-0.8} \propto (N^3 \cdot d^2)^{-0.4} \tag{4}$$

$$a \text{ and } \delta \propto N^{1.2} \cdot d^{0.8} \propto (N^3 \cdot d^2)^{0.4} \tag{5}$$

The results regarding d_{32}, published by various authors (13–18), were compared with those of this work. Our results for the exponents of the impeller speed (N) and impeller diameter (d) agreed well with the results of several other researchers.

P_V, the stirring consumption power per unit of liquid volume, was calculated under turbulent flow conditions with a correlation equation reported by Rushton, Costich, Everett (19) and Laity and Treybal (20).

$$P_v = \frac{(4 \cdot K_1 \cdot \rho_c \cdot N^3 \cdot d^5)}{(g_c \cdot \pi \cdot D^3)} \tag{6}$$

$K_1 = 6.3$ for 6 flat-blade turbines under turbulent stirring conditions. In the case of geometrically similar tanks, Eq. (6) can be expressed as follows:

$$P_V \propto N^3 \cdot d^2 \tag{7}$$

Equations (8 and 9) can be derived from Eqs. (4, 5, and 7) because the flow pattern of fluid in the stirred tank was turbulent within the range of the experimental conditions ($1.51 \times 10^4 < \mathrm{Re}[= (d^2 \cdot N \cdot \rho_c)/\mu_c] < 1.49 \times 10^5$) for this work.

$$d_{32} \propto P_V^{-0.4} \tag{8}$$

$$a \text{ and } \delta \propto P_V^{0.4} \tag{9}$$

From Eqs. (8 and 9), we know that a and δ (or d_{32}) were equal when P_V was kept constant using geometrically similar tanks.

Figure 2 shows the relationship between d_{32} and the Weber number in 2 different sized tanks. Figure 2 was also plotted using the results calculated from Calderbank's empirical equation (13). As shown in Fig. 2, experimental data of this work show a straight line, irrespective of tank size, and the results of this work agreed fairly well with those of Calderbank. The values for d_{32} obtained from tank I and tank II at $P_V = 341$ kg/m^2·s, calculated from Eq. (6), were 1.92×10^{-4} m and 1.99×10^{-4} m, respectively. From these results, we confirmed that P_V was

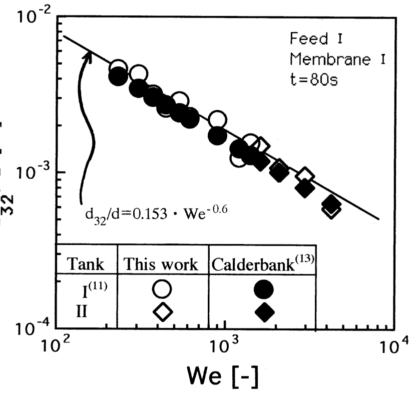


Figure 2. Plot of d_{32} vs. We.

applicable to the scale-up standard for the O/W emulsion droplet sizes. The volume fractions for the inner oil phase in emulsion, ϕ_o, obtained by stirring for 80 seconds with tanks I and II under constant impeller speed ($N = 10\ s^{-1}$), were used for calculating δ. The values of ϕ_o obtained with tank I and tank II were 0.375 and 0.315, respectively. The values of δ in tank I and tank II calculated from Eq. (3) using the values of ϕ_o were 2.74×10^{-5} m and 1.89×10^{-5} m, respectively. We can confirm that the value of δ in our work nearly agrees with that of Goswami and Rawat (21), if the value of d32 calculated by this work is used instead of the value assumed by Goswami. The values of a in tank I and tank II, calculated from Eq. (2), were $3.05 \times 10^3\ m^{-1}$ and $5.02 \times 10^3\ m^{-1}$, respectively. Though the emulsion was under constant stirring speed, the value of δ calculated in tank II was lower than that calculated in tank I; in contrast, the value of a was higher. These results can be explained by the differences in dissipation energy between tank I and tank II. The value of a was higher because the value of d_{32} in tank II decreased with increasing dissipation energy.

The values of a and δ calculated for the emulsion made by (Feed I and Membrane I) were used for calculating the mass transfer coefficients independently from the material systems and contact times studied.

Scale-Up for the Mass Transfer Coefficient

The time course curves for hydrocarbons concentrations (toluene, heptane) in the inner and outer oil phases obtained at constant impeller speed ($N = 10\ s^{-1}$) for tank I and II are shown in Fig. 3. The permeation rate of hydrocarbon components through the liquid membrane depends on the diffusivity and, in particular, the solubility of the hydrocarbon through the liquid membrane (8). Thus, the toluene concentration in the outer oil phase was higher than that of heptane because the permeation rate of toluene through the liquid membrane was higher than that of heptane. The toluene concentration in the outer oil phase increased with increasing contact time. Conversely, the toluene concentration of the inner oil phase decreased with increasing contact time. The heptane concentration in the inner and outer oil phases increased with increasing contact time. This tendency indicated that the permeation rate of heptane is much smaller than that of toluene. In addition, when the toluene and heptane concentrations in the outer oil phase were compared between tank I and tank II, the concentrations obtained from the larger tank (tank II) were higher than those obtained from the smaller tank (tank II). This observation implies that smaller O/W emulsion droplet sizes are obtained with the larger tank when impeller speed is kept constant. Accordingly, the permeation rates of toluene and heptane in tank II were higher than those in tank I. The heptane concentration in the inner oil phase in tank I was lower than in tank II. This tendency can be explained by the difference in solubilities of hydrocarbons

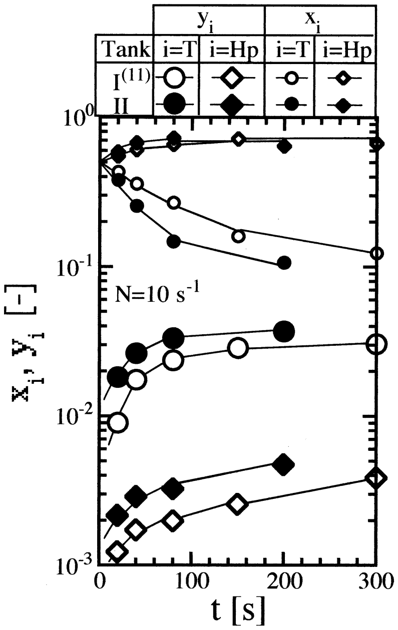


Figure 3. Time course curves for the concentration of each component in the inner oil phase and the outer oil phase.

through the liquid membrane and the difference in the dissipation energy for the 2 tank sizes.

The O/W emulsion and the solvent were the dispersed and the continuous phases, respectively, throughout the experiment. The total rate of mass transfer for the permeable component consists of the rates of permeation, the membrane breakage, and the mechanical entrainment. Kawasaki et al. (22) reported that the mass transfer rate was nearly equal to that calculated from the membrane permeation alone, which was within the stirring times used in this work. Therefore, the effects of membrane breakage and mechanical entrainment on mass transfer of the permeable component were deemed negligible in this work. We assumed that the mass transfer of each component occurred only by membrane permeation and that the mass transfer resistance might depend greatly on the dispersed phase (because the distribution coefficient of each component within a liquid membrane was very small within the entire material system used for this experiment). The transfer rate of component *i* was calculated by using Eq. (10) as follows (11):

$$\frac{d\,(E\cdot y_i)}{dt} = k_{\mathrm{d},i}\cdot a\cdot(x_i - y_i)\cdot V \tag{10}$$

$k_{d,i}$ can be calculated from the slopes at each stirring time from the time course curves for the permeated amount of hydrocarbon component i and the values of a obtained from Eq. (2). The relationship between $k^*_{d,i} = (k_{d,i} \cdot (\delta) / (\rho_w \cdot D_i \cdot H_i)$ and $t^* = (t \cdot D_i)/\delta^2$ in 2 different tanks is shown in Fig. 4. Where $k^*_{d,i}$ is the ratio of $k_{d,i}$ obtained from each tank to $k_{d,i}$ of an ideal one-dimensional plate liquid membrane and and t^* is a dimensionless stirring time. The diffusivity of component i within a liquid membrane, D_i, was calculated with the equation of Wilke and Chang (23). McAuliffe's experimental values (24) were used as the distribution coefficients of membrane I, and experimental values from our work were used as those of membrane II. $k^*_{d,i}$ was proportional to $t^{*-0.5}$ and were independent of tank size and material systems used. Although our results agree with the film penetration theory, the reasons for this result are not yet clear. Explaining these results will require further study.

The relationship between $k^*_{d,i}/t^{*-0.5}$ and H_i in the 2 different tanks is shown in Fig. 5. The values of $k^*_{d,i}/t^{*-0.5}$ for each component were equal irrespective of the tank sizes used. Also, the results of all the test runs, regardless of tank and material system used, were within approximately ±30 % error and lay on the same straight line. The slope of this straight line was–0.5. This linearity, as stated in the

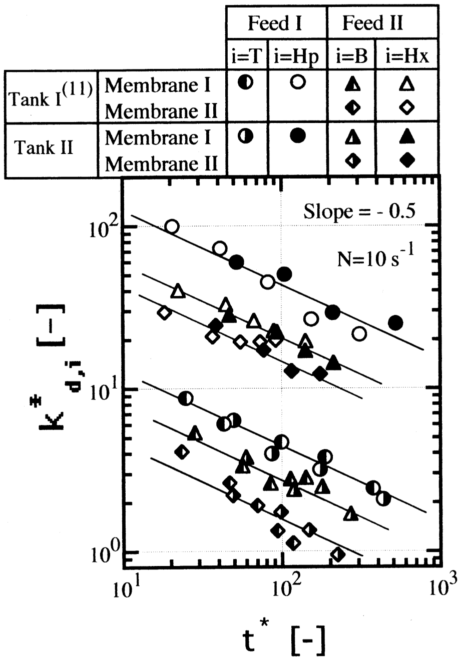


Figure 4. Plot of $k^*_{d,i}$ vs. t^*.

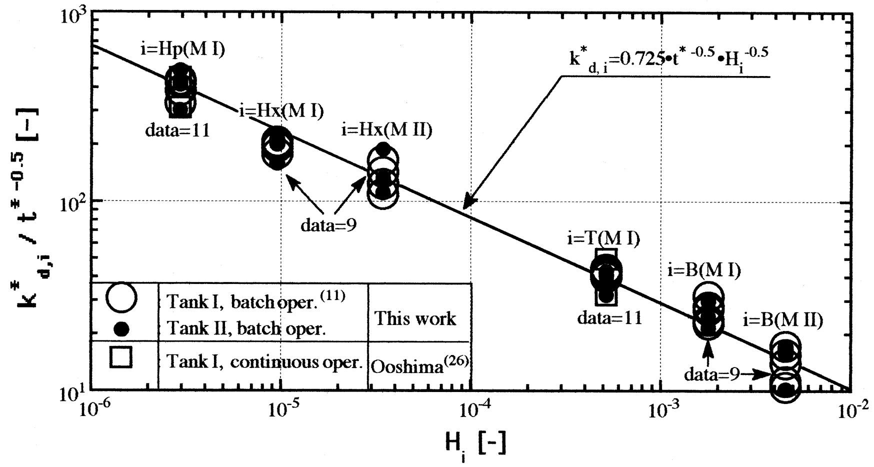


Figure 5. Plot of $k^*_{d,i}/t^{*-0.5}$ vs. H_i. M I represents Membrane I, and M II represents Membrane II.

previous paper (11), might be attributed to a composite of factors: the characteristic concentration distribution of bulk separation that was formed within emulsion (25), additions of surfactant and permeation promoter into the membrane solution, the solubility effects of various coexisting hydrocarbon components, membrane breakage, and mechanical entrainment. Figure 5 also shows the results of Ooshima, Kato, and Kawasaki (26) of a continuous operation, in which the same size of stirred tank (tank I) and material system (Feed I, Membrane I) was used as the batch operation in this work. Ooshima, Kato, and Kawasaki obtained $k^*_{d,i}$ values that could be calculated by corresponding the mean residence time of the continuous operation to a stirring time of the batch operation. The results of the present work agree fairly well with that of their previous work on continuous operation.

From the above results, regardless of tank sizes and material systems used, a simplified correlation equation was obtained for the mass transfer coefficient of the dispersed phase.

$$
\begin{aligned}
&k^*_{d,i} = 0.725 \cdot t^{*-0.5} \cdot H_i^{-0.5} \\
&1.0 \times 10 < t^* < 6.0 \times 10^3 \qquad (11) \\
&2.0 \times 10^{-6} < H_i < 5.0 \times 10^{-3}
\end{aligned}
$$

From Eq. (11), obtained through calculating Eq. (2) for a and Eq. (3) for δ, we knew that the mass transfer coefficients for the dispersed phase were equal, irrespective of tank sizes at constant P_V when geometrical similarity of the tanks was preserved. We also knew that the properties between emulsion and solvent were unchanged. The results of the scale-up described in this work will require investigation into their practical applications. When a stirred tank of a practical scale was employed as a contacting apparatus, $k_{\mathrm{d},i}$ was successfully estimated from Eq. (11). Therefore, the purity of the material separated by the countercurrent multistage and the energy required for separation could be forecasted on a practical scale.

CONCLUSION

1. Independent of tank size, a sauter mean diameter of the O/W emulsion dispersed in the O/W/O emulsion was successfully correlated with the Weber number. The results of this work agreed well with those of previous reports.
2. The mass transfer coefficient of hydrocarbon through the O/W emulsion could be correlated with the dimensionless stirring time and the solubility of the membrane phase, irrespective of tank size and material systems (hydrocarbon feeds, aqueous membrane solution). The result of the batch operation in this work could be successfully applied to a continuous operation system.
3. We suggest that the stirring consumption power per unit of liquid volume in the tank is applicable as the scale-up standard for obtaining the mass transfer coefficient of hydrocarbon through the O/W emulsion when geometrical similarity of tanks is preserved and the properties between emulsion and solvent are not changed.

NOMENCLATURE

a	specific interfacial area between dispersed phase and continuous phase (m^{-1})
$\mathrm{C_S}$	mass fraction of saponin in aqueous membrane phase (−)
$\mathrm{C_{SUL}}$	mass fraction of sulfolane in aqueous membrane phase (−)
D	stirred tank diameter (m)
D_i	diffusivity of component i in aqueous membrane phase (m^2/s)
d	impeller diameter (m)
d_i	size of bin i (m)
$\mathrm{d_{32}}$	sauter diameter of dispersed phase (m)
E	mass of continuous phase (kg)
g_c	gravitational constant
H	liquid depth or tank high (m)
H_i	distribution coefficient of component i in aqueous membrane phase (−)

K_1	proportionality constant for power
$k_{\mathrm{d},i}$	dispersed phase mass transfer coefficient of component i (kg/s·m^2)
$k^*_{\mathrm{d},i}$	dimensionless dispersed phase mass transfer coefficient of component i [$= (\mathrm{k}_{\mathrm{d},i}\cdot\delta)/(\rho_\mathrm{W}\cdot D_i\cdot H_i)$] (−)
N	impeller speed (s^{-1})
n_i	number of drops in bin i (−)
P	stirring power (kg·m/s)
P_V	stirring consumption power per unit liquid volume in the stirred Tank (kg/m^2·s)
Re	impeller Reynolds number [$= (d^2\cdot N\cdot\rho_\mathrm{c})/\mu_\mathrm{c}$] (−)
T	operating temperature (°C)
t	contact time (seconds)
t^*	dimensionless contact time [$= (t\cdot D_i)/\delta^2$] (−)
V	volume of total liquid or tank (m^3)
We	weber number [$= (N^2\cdot d^3\cdot\rho_\mathrm{d})/\sigma$] (−)
x_i	mass fraction of component i in dispersed phase (−)
y_i	mass fraction of component i in continuous phase (−)

Greek

δ	thickness of aqueous membrane phase (m)
ϕ_d	volumetric fraction of dispersed phase (−)
$\phi_{\mathrm{o},0}$	volumetric fraction of inner oil phase in dispersed phase at initial (−)
ϕ_o	volumetric fraction of oil phase in dispersed phase (−)
μ_c	viscosity of continuous phase (kg/m·s)
ρ_c	density of continuous phase (kg/m^3)
ρ_d	density of dispersed phase [$=\rho_\mathrm{o}\cdot\phi_\mathrm{o} + \rho_\mathrm{w}\cdot(1-\phi_\mathrm{o})$] (kg/m^3)
ρ_W	density of aqueous membrane phase (kg/m^3)
σ	interfacial tension between dispersed phase and continuous phase (kg/s^2)

Subscript

B	benzene
c	continuous phase
d	dispersed phase
Hp	*n*-heptane
Hx	*n*-hexane
i	component i
j	component j

S	saponin
SUL	sulfolane
T	toluene
w	aqueous membrane phase
o	oil phase
0	at initial (t = 0)

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