Separation Science and Technology

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Kawasaki, Junjiro , Kosuge, Hitoshi , Egashira, Ryuichi and Asawa, Tomotake(2009) 'Mechanical Entrainment in W/O/W Emulsion Liquid Membrane', Separation Science and Technology, 44: 1, 151 — 168

To link to this Article: DOI: 10.1080/01496390802437115

URL: http://dx.doi.org/10.1080/01496390802437115

Separation Science and Technology, 44: 151–168, 2009
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ISSN: 0149-6395 print/1520-5754 online
DOI: 10.1080/01496390802437115

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Mechanical Entrainment in W/O/W Emulsion Liquid Membrane

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Abstract: An experimental study of mechanical entrainment in W/O/W emulsions is conducted. W/O/W emulsions are stirred for various stirring times under the conditions that mechanical entrainment solely occurs, and changes in volume of the W/O emulsions and size distribution of the internal water droplets are measured. The rate of change in number of the water droplets entrained is found to be proportional to the volume fraction of W/O emulsions. Based on this result, a new model for mechanical entrainment is developed. The calculated change in W/O emulsion volume with time agrees with the observed ones except in the region near phase inversion. Then, phase inversion is discussed.

Keywords: Emulsion liquid membrane, mechanical entrainment, phase inversion, W/O/W emulsion

INTRODUCTION

Emulsion liquid membrane (ELM) techniques, which provide large mass transfer rates, have been expected as promising powerful separation methods. So far, there have been many studies on the application of the ELM techniques to separation processes, such as separation of metallic ions

Received 20 January 2008; accepted 10 July 2008.

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from wastewater (1), antibiotics from aqueous solutions (2–4), and aromatics from aliphatics (5). However, practical applications of the ELM techniques are not so many, because a state of emulsion changes during operation by the phenomena such as breakage of membrane, permeation, and mechanical entrainment, and it practically makes separation performance reduced.

Breakage of the membrane phase in three-phase emulsions, which is caused by friction of the external phase or swelling of the membrane phase (6), causes leakage of the internal phase, and consequently reduces recovery factors of ELM processes. According to the previous works (7,8), breakage of membrane in W/O/W emulsions can be suppressed by adding more surfactant than a critical amount. Meanwhile, permeation and mechanical entrainment of the external phase dilute the internal phase, so that those phenomena reduce enrichment factors of ELM processes. Permeation of water in W/O/W emulsions, which is caused by concentration difference between the internal and external aqueous phases (9), can be suppressed by decreasing the concentration of surfactant (8,10), and increasing the volume of the internal aqueous phase (8,10). Similarly, mechanical entrainment can be suppressed by increasing the volume of the internal phase (10,11), and decreasing the surfactant concentration (8,10–12), initial volume fraction of W/O emulsions (12), and stirring speed (11,12). As for the mechanism of mechanical entrainment, Ding and Xie (13) proposed an occlusion model that emulsion globules occlude and take in the external phase. If this phenomenon occurs, the size of water droplets taken in may change with time, because W/O emulsions swell. However, there have been few studies on the effect of size and number of the internal droplets on the mechanical entrainment.

On the other hand, phase inversion has been studied by many researchers (14–16). According to Marianna Rondon-Gonzalez et al. (16), when viscosity of the oil phase is higher than that of the water phase, O/W emulsion is inverted to W/O emulsion through the formation of W/O/W, where the apparent volume of the dispersed phase increases until a critical value is reached and the inversion is triggered. On the other hand, if the water phase is more viscous than the oil phase, the inversion occurs through the formation of a pseudo-fibrous structure, in which the connectivity of the internal phase is produced by drop elongation.

In the present work, to study the mechanism of mechanical entrainment, W/O/W emulsions are stirred for various stirring times under the conditions that mechanical entrainment solely occurs, and volume of the W/O emulsions, and the number and size of the internal water droplets are measured at different stirring times. Based on the results, a new model of mechanical entrainment is developed, and the mechanisms of mechanical entrainment and phase inversion are discussed.

EXPERIMENTAL APPARATUS AND PROCEDURES

An apparatus used is a glass-made stirred vessel of 0.092 m in diameter, equipped with four baffle plates of 0.01 m in width and a six-flat blade disk turbine of 0.05 m in diameter. The apparatus is immersed in a constant temperature bath to keep solution temperatures constant. A heptane solution containing a given amount of sorbitan monooleate (Span 80) is used as a membrane phase. To prepare W/O emulsions, the heptane solution is stirred in ion exchange water with a homogenizer at a constant speed. Meanwhile, as the external phase in W/O/W emulsion, ion exchange water is used to avoid permeation.

The experiments on W/O/W emulsion are carried out as follows. The W/O emulsions prepared are stirred in ion exchange water with the impeller at a constant speed for a given time. If phase inversion occurs during the stirring, the occurrence time of phase inversion is recorded. After the stirring is stopped, the motion of the continuous and dispersed phases is observed until completion of the phase separation, to determine the type of emulsion. Then, the following quantities are measured: volume of the continuous and W/O emulsion phases, and size distribution of the W/O emulsions and internal droplets. The size of internal water droplets is measured by a particle size analyzer having the upper limit of 60 μm in diameter. In the experiments at the standard condition, a small amount of W/O/W emulsions is sampled during the stirring, and put on the surface of water containing 3% saponin to take microphotographs. The same operations are done for the W/O emulsions after phase separation, to take microphotographs of them. The above experiments are carried out with different stirring speeds, heights of the impeller, initial volume fractions of W/O emulsions, and stirring time. The experimental conditions are shown in Table 1.

Emulsion types of two-phase and three-phase emulsions are determined by motion of the continuous and dispersed phases during phase separation and clarity of the aqueous and oil phases after phase separation. In order to find the criteria for emulsion types, demulsification of heptane-water emulsions was conducted without surfactants. The settling and coalescing interfaces in W/O emulsions moved faster than those interfaces of O/W emulsions. The aqueous and oil phases in W/O emulsions were clear, but the aqueous phase in O/W emulsions was cloudy. Meanwhile, the three-phase emulsions in the present study contained milky droplets during the stirring, and the continuous and dispersed phases became clear after phase separation. When phase inversion occurred, the entire liquid becomes cloudy. These observations indicate that W/O/W emulsions are formed by stirring and changed into W/O emulsions at phase inversion.

Table 1. Experimental conditions

Internal and external phase	Ion exchange water
Membrane phase	*n*-heptane with SPAN 80 (Volume fraction, $C_S = 0.05$)
Volume fraction of internal aqueous phase in W/O emulsion, ϕ_{in}	0.5
Volume fraction of W/O emulsion, ϕ_{WO}	0.0625, 0.125*
Total volume of solution, $V_T[m^3]$	4×10^{-4}
Height of impeller, $H^*[-]$	0.353, 0.50*, 0.638
Stirring time, t[s]	30–5000
Stirring speed, $N[s^{-1}]$	7.5, 8.3*, 10.0, 11.7
Temperature, [K]	298

*Represents the standard experimental condition.

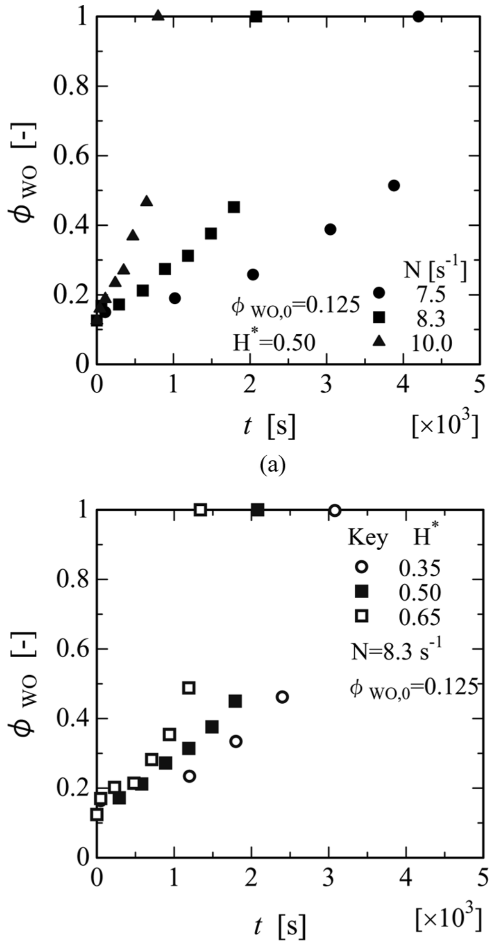
EXPERIMENTAL RESULTS

Changes in Volume of W/O Emulsion with Stirring Time

In the present study, the internal and external aqueous phases are pure water. Thus, no water permeation occurs, while whether breakage of membrane occurs cannot be examined by the present experiments. Then, in order to consider a possibility of breakage of membrane a number of molecular layers of surfactant on the oil-water interface (7,8) was calculated under the assumptions that all the surfactant molecules were spherical and adsorbed on the surface of the oil phase. The number of the surfactant layers was larger than 2 of the critical value (8) for all the experiments. Thus, the authors assume that breakage of membrane does not occur, and consequently mechanical entrainment solely occurs in the present experiments.

Figure 1 shows changes in volume fraction of W/O emulsion with stirring time. The parameters in Figs. 1(a) and 1(b) are stirring speed and height of the impeller, respectively. The volume fraction of W/O emulsion increases with stirring time, and phase inversion ($\phi_{WO} = 1$) occurs at a certain time. As the stirring speed and height of the impeller increase, the slopes of the curves become steeper and phase inversion occurs earlier. When the initial values of volume fraction of W/O emulsion increased, slopes of the curves also became steep.

As for mechanical entrainment, Fujinawa et al. (10) carried out the experiments of W/O/W emulsion with relatively low stirring speeds, and reported that mechanical entrainment only occurs at early stages



(a)

(b)

Figure 1. Change in volume fraction of W/O emulsion with stirring time at different (a) stirring speeds and (b) heights of impeller.

of stirring where W/O/W emulsion is just forming. However, in the region except the vicinity of phase inversion in Fig. 1, the slopes of the curves increase regularly with increase in stirring time, that is, mechanical entrainment occurs throughout the stirring time. This is because the stirring speed in the present study is larger than that in the experiment by Fujinawa et al. (10). However, a change in volume fraction at the first measuring point at t = 60s in Fig. 1 is larger than a change at the second measuring point for each run. This may indicate that another phenomenon occur with mechanical entrainment at the early stage of stirring, because W/O/W emulsion is rapidly formed in the region and the area of the oil-water interface increases. In the last region near phase inversion, the volume fraction of W/O emulsion exceeds 0.5 and slopes of the curves become steep. Since the continuous phase becomes small in quantity, another phenomenon might occur with mechanical entrainment.

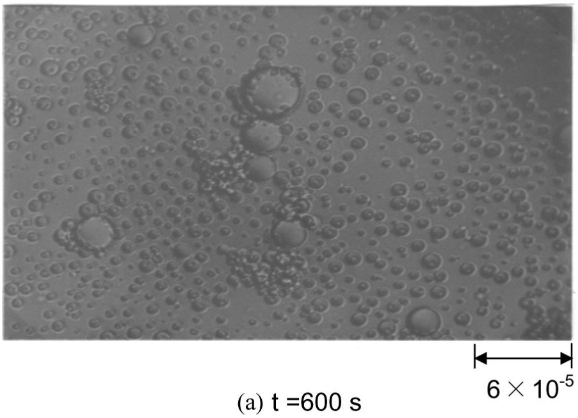
Changes in Size and Number of the Internal Water Droplets with Stirring Time

Figure 2 shows microphotographs of internal water droplets at $t = 600$s, 1800s, and 2200s (phase inversion), of which the experiment is made under the standard condition shown in Table 1. As stirring time increases, the internal water droplets become large, and the number of large water droplets increases. In order to show the relationship between the size and the volume of internal water droplets, the size distribution curves of the internal water droplets are shown in Fig. 3. The vertical axis is the volume fraction of the water droplet of d_{in} in diameter, and the horizontal axis is the diameter of a water droplet, of which the upper limit of 60 μm is given by the detectable upper limit of the particle analyzer. As stirring time increases, the diameter at the peak of a curve becomes large, and large droplets increase in number. Compared with the curve at $t = 1800$s, the curve at $t = 2200$s is close but the number of water droplets of around 60 μm in diameter increases. Furthermore, water droplets of more than 60 μm in diameter are observed more in a microphotograph at $t = 2200$s.

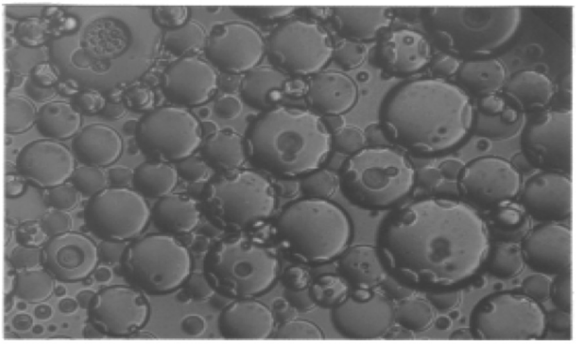
Assuming that internal water droplets are spherical, the number density of the internal water droplet of d_{in} in diameter, $n_{in,d}$ can be evaluated by

$$n_{in,d} v_{in,d} = F_{V,d} V_{in} \tag{1}$$

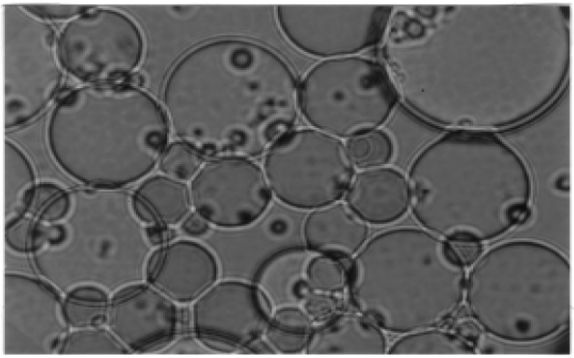
where V_{in} is volume of the internal aqueous phase per unit volume of W/O/W emulsion at a given stirring time, $F_{V,d}$ is the volume fraction of the internal water droplet of d_{in} in diameter, and $v_{in,d}$ is volume of a droplet of d_{in} in diameter. Figure 4 shows the distribution curves of the number density of internal water droplets at different stirring times. Since the size distribution curves of internal water droplets are convex upwards as shown in Fig. 3, the distribution curve of the number density of internal water droplets should be convex upwards but they are the downward-sloping curves as shown in Fig. 4. This may be due to the resolution of the particle size analyzer, and the detectable minimum size of 5 μm is a little coarse.



(a) t =600 s



(b) t =1800 s



(c) t =2200 s

Figure 2. Internal water droplet in W/O emulsion at (a) t = 600s (b) t = 1800s (c) t = 2200s.

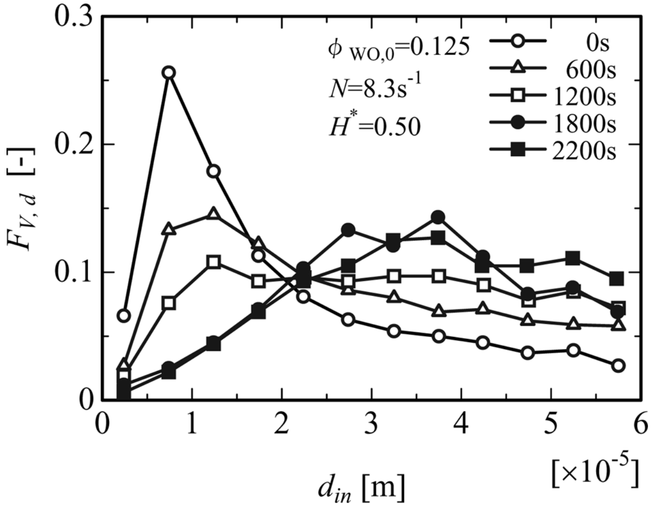


Figure 3. Size distribution curve of internal water droplet at different stirring times.

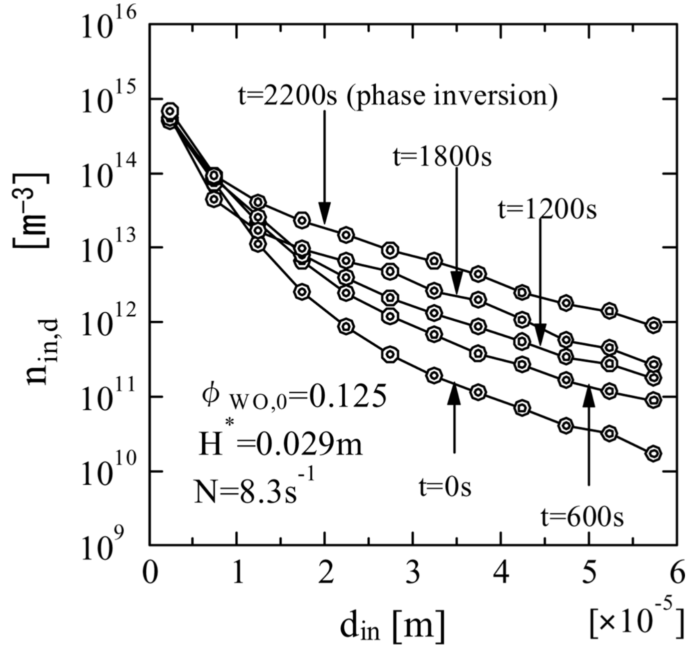


Figure 4. Number density of internal water droplet.

Nevertheless, Fig. 4 shows a reasonable tendency that the number density becomes lower as the droplet diameter increases and stirring time decreases.

Changes in Number of the Entrained Water Droplets with Stirring Time

The volume of the entrained water droplet of d_{in} in diameter per unit volume of W/O/W emulsion can be calculated by

$$(n_{in,d} - n_{in,d,0})V_{in,d} = F_{V,d}V_{in} - F_{V,d,0}V_{in,0} \tag{2}$$

where the subscript 0 represents the beginning of stirring, and the observed values in the right hand side are used to calculate the volume of the entrained water droplets. Figure 5 shows the volume distribution curves of the water droplets entrained, of which the data are obtained at the standard condition. In the range of $0 < t < 1200$s where the volume fraction of W/O emulsion is less than 0.3 as shown in Fig. 1, water droplets of various sizes are uniformly taken in. After the time ($t > 1200$s), the large water droplets gradually increase. Meanwhile, the volume of the water droplets entrained increases with increase in stirring time at any diameter.

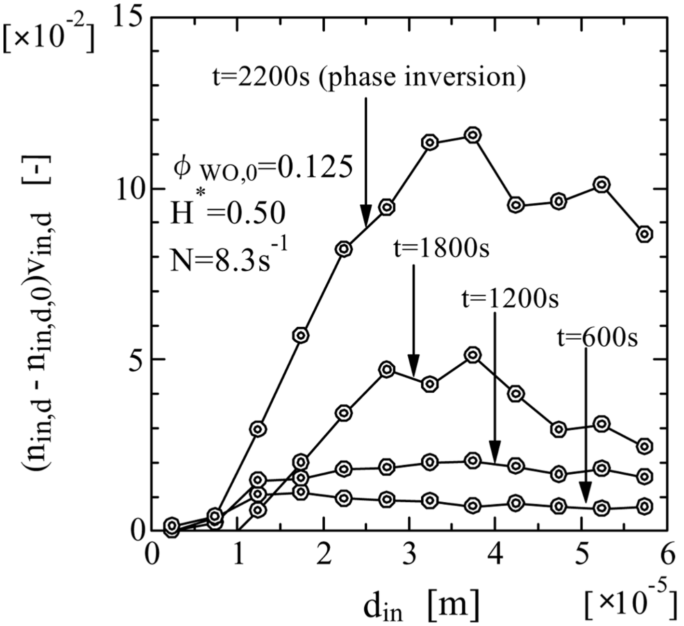


Figure 5. Volume distribution curve of water droplet entrained.

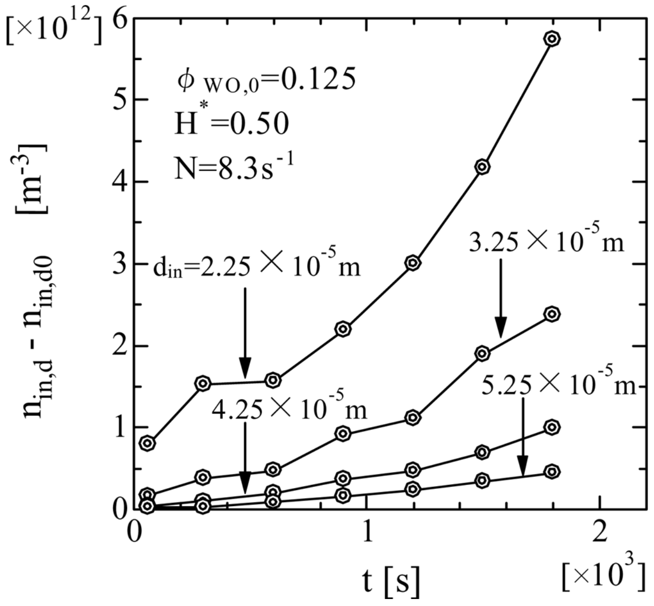


Figure 6. Change in number density of water droplet entrained with stirring time.

Figure 6 shows changes in the number density of water droplets entrained with stirring time are shown. In the figure, the data of $d_{in} \geq 22.5\mu m$ are shown because the number density of the smaller droplets has a little large experimental error. The number of water droplets entrained increases with increase in stirring time. Furthermore, the slopes of the curves become larger with stirring time, and with decrease in diameter. Similar tendencies were observed for other data. Since the volume fraction of W/O emulsion increases with stirring time as shown in Fig. 1, a rate of change in the number density of water droplets entrained may be affected by the volume fraction of W/O emulsion. In order to confirm this, the rates of change in the number density of water droplets entrained, $dn_{in,d}/dt$, is obtained at each stirring time. The results are shown in Fig. 7. The rates of change in the number density of water droplets entrained are roughly proportional to the volume fraction of W/O emulsion, and increase with decrease in diameter. Similar results were obtained for other experimental data.

MODELING OF MECHANICAL ENTRAINMENT

Figure 7 indicates that a rate of change in the number density of water droplets entrained can be expressed by

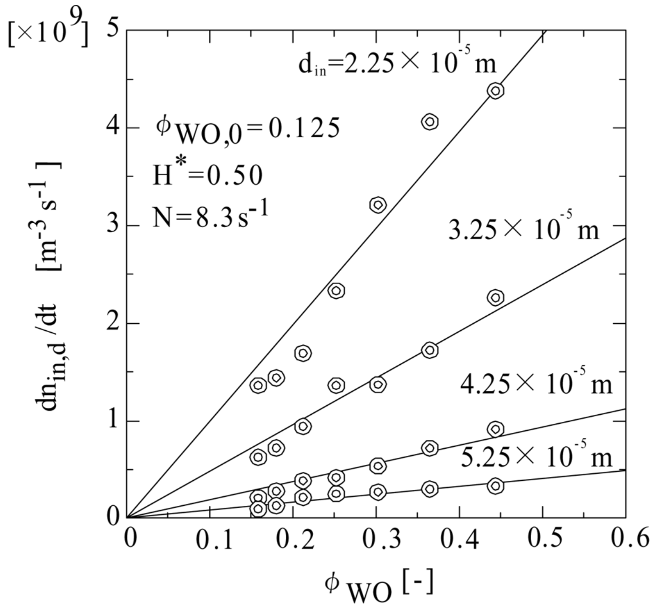


Figure 7. Effect of volume fraction of W/O emulsion on rate of entrainment.

$$\begin{aligned} &\frac{dn_{in,d}}{dt} = k\phi_{WO} \\ &t = 0; n_{in,d} = n_{in,d,0}; \quad \phi_{WO} = \phi_{WO,0} \end{aligned} \tag{3}$$

Since a proportional constant, k, is affected by the diameter of the water droplets entrained, a relationship between the two is shown in Fig. 8. The values of k at each experimental condition decrease linearly with increase in droplet diameter, and the slopes of the lines are roughly the same. Then, the values of k are correlated in terms of diameter of water droplets. The correlation equation obtained is expressed as

$$k = \exp(k_0 - 7.78 \times 10^4 d_{in}) \tag{4}$$

As shown in Fig. 8, the values of k at a given diameter are affected by stirring speed and height of the impeller and less affected by initial volume of W/O emulsion. Therefore, k_0 in Eq. (4) is a function of the first two variables. Then the values of k_0 are correlated with N and H, so that the following equation is obtained.

$$k_0 = 15.32 H^{*0.0318} N^{0.238} \tag{5}$$

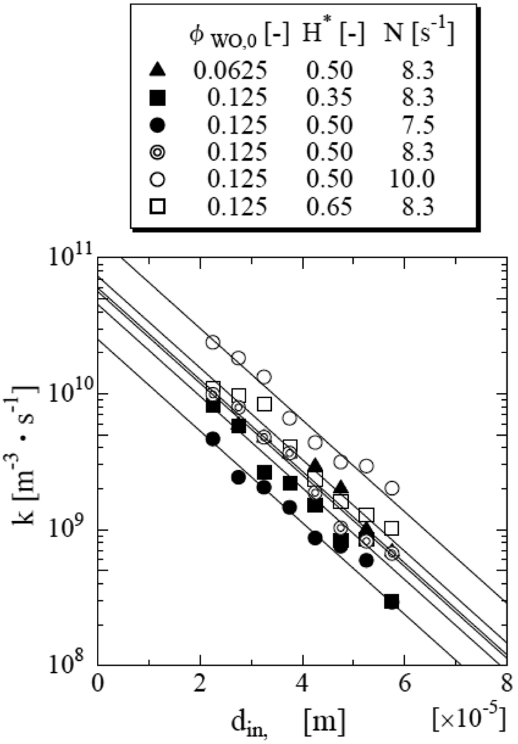


Figure 8. Effect of diameter of water droplet entrained on the rate constant *k*.

In order to confirm the validity of the model expressed by Eq. (3), the volume fraction of W/O emulsion at $t = t_n$ is calculated by the following equation.

$$\phi_{WO}(t = t_n) = \phi_{WO}(t = t_{n-1}) + \left\{ \sum_{d_{in}} k V_{in,d} \phi_{WO}(t = t_{n-1}) \right\} (t_n - t_{n-1}) \quad (6)$$

In numerical calculation of Eq. (6), $0 < d_{in} \leq 60\mu m$ and $t_n - t_{n-1} = 10s$ are used. The calculated volume fraction of W/O emulsion is shown in Fig. 9 with the observed data. Although the model equation is very simple and the proportional constant, *k*, is determined by the data of $d_{in} \geq 22.5\mu m$, the calculated volume fractions of W/O emulsion agree with the experimental data except the region of the beginning of stirring and the region near phase inversion. This result indicates the validity of the present model for mechanical entrainment because there is a possibility of another phenomenon in the two regions.

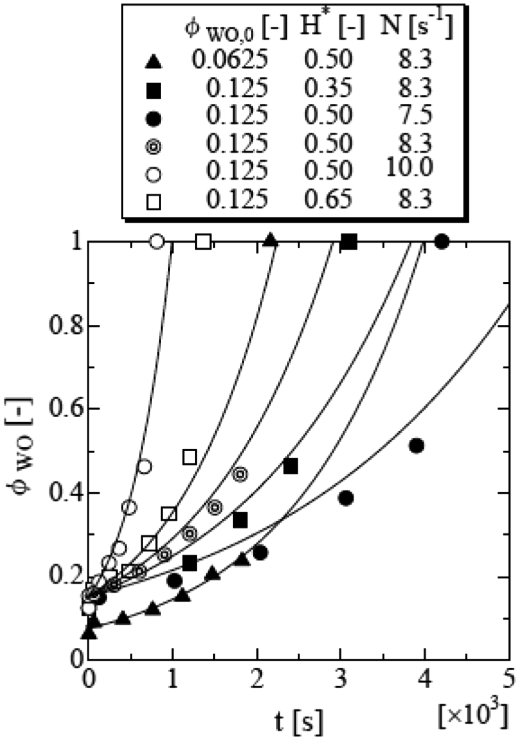


Figure 9. Comparison of calculated volume fraction of W/O emulsion with observed ones.

Meanwhile, the volume fraction of the water droplet of d_{in} in diameter, $F_{V,d}$, at $t = t_n$ is expressed by

$$F_{V,d} = \frac{kv_{in,d}\phi_{\mathrm{WO}}(t = t_n)}{\sum\limits_{d_{in}} kv_{in,d}\phi_{\mathrm{WO}}(t = t_n)} = \frac{kv_{in,d}}{\sum\limits_{d_{in}} kv_{in,d}} \tag{7}$$

When the stirring speed, the height of the impeller, and the diameter of the water droplet are specified, the value of k, which is determined by Eq. (4) and (5), becomes constant with respect to stirring time. Therefore, the size distribution curve of the water droplet entrained becomes unique without respect to stirring time. The size distribution curve obtained is shown in Fig. 10 with the observed distribution curve at $t = 0$s, 1800s and 2200s. The water droplets having the calculated size distribution curve are constantly entrained into W/O emulsions. A globule of W/O emulsion

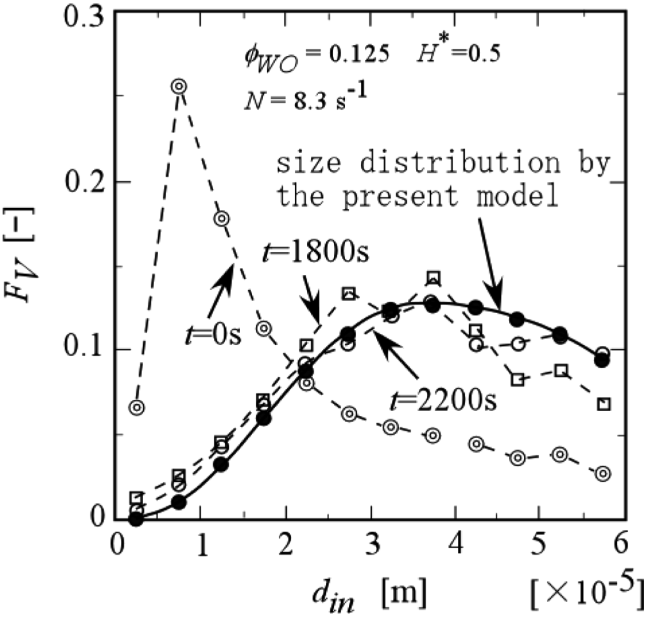


Figure 10. Size distribution of water droplets entrained.

having a certain diameter entrains water droplets in a certain range of diameters, however, since the unit volume of W/O/W emulsion contains many globules of W/O emulsions having various sizes, such entrainment may be possible. As entrainment of water droplets having the calculated size distribution proceeds, the location of the peak of size distribution curve of internal water droplets shifts to the right. At $t = 1800$s, the observed size distribution curve of the internal water droplets becomes close to that of the water droplets entrained, and the observed size distribution curve at $t = 2200$s agree with the calculated curve.

Those results indicate that the present model for mechanical entrainment is valid. However, Fujinawa et al. (10) reported that the amount of mechanical entrainment increases with increase in the concentration of surfactant. Ding and Xie (13) mentioned the effects of the kind of surfactant and viscosity on entrainment. Therefore, the constant values in Equation (3) may be affected by the surfactant concentration, kinds of surfactant, and the viscosity and surface tension of the W/O emulsion. Further experiments with different surfactants and membrane phases are necessary to obtain the more general model for mechanical entrainment.

MECHANICAL ENTRAINMENT AND PHASE INVERSION

Figure 10 may give an impression that phase inversion is caused by mechanical entrainment solely. As shown in Fig. 9, however, the occurrence-time of phase inversion that is predicted by the present model is quite different from the observed one, and a rate of change in volume fraction of W/O emulsion near phase inversion is also different from the observed data. In

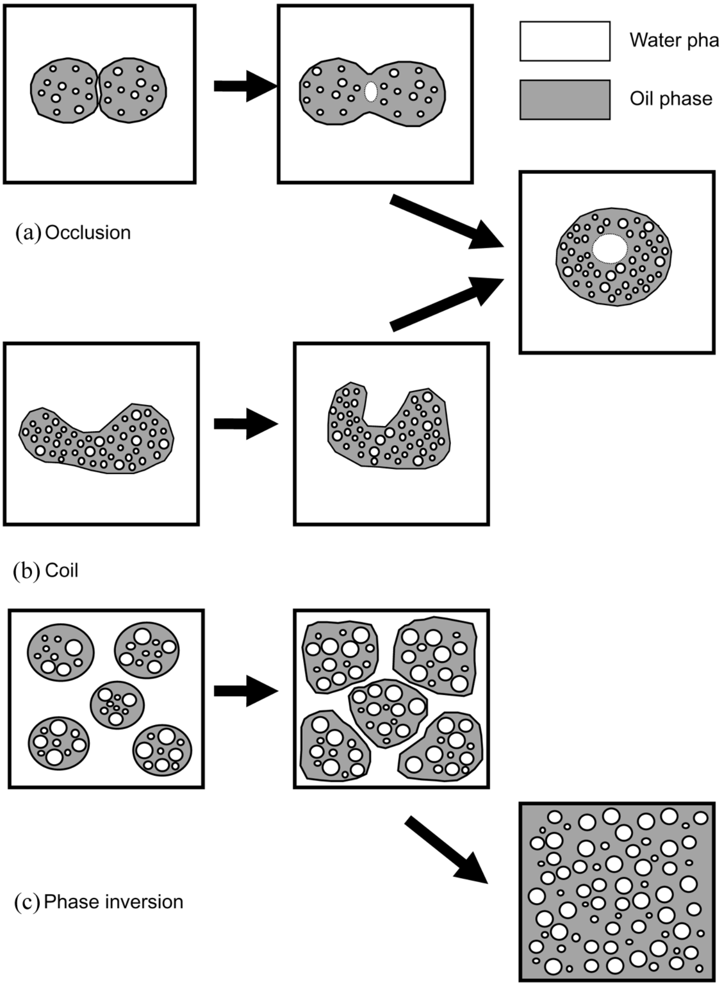


Figure 11. Schematic illustrations for mechanical entrainment and phase inversion.

the photographs of the internal water droplets at phase inversion, the large water droplets of 60 μm or more in diameter are observed. Therefore, another phenomenon as well as mechanical entrainment may occur at phase inversion.

As Ding and Xie (13) stated, mechanical entrainment occurs by occlusion of W/O emulsions. The external water is entrapped in a manner shown in Fig. 11(a). If concave-shaped emulsions are formed, occlusion may easily occur (12), and entrainment of the external water might be possible in a manner shown in Fig. 11(b). When the volume of W/O emulsion further increases, the shape of W/O emulsion becomes distorted (17) and the continuous phase surrounded by W/O emulsions gradually becomes thin. If a thin film of the continuous phase is torn at a spot, several globules of W/O emulsion coalesce with each other while taking a considerable amount of the continuous phase in, as shown in Fig. 11(c). Consequently, the volume of W/O emulsion abruptly increases. Near phase inversion, breakage of a thin film of the continuous phase occurs everywhere, and causes an abrupt change in volume of the W/O emulsion.

CONCLUSIONS

In order to study mechanical entrainment in W/O/W emulsions, W/O emulsion is stirred with ion exchange water at the conditions that mechanical entrainment solely occurs, and the volume of W/O emulsion and size distribution of internal water droplets are measured. As stirring time increases, the size of internal water droplets increases, and the number of large water droplets also increases. Those increases are caused by mechanical entrainment of the external aqueous phase, and the size and number of the water droplets entrained increase with increase in stirring time. The rates of change in number of the water droplets entrained are found to be proportional to the volume fraction of W/O emulsion. Base on the experimental results, a new model for mechanical entrainment is developed, and the volume fraction of W/O emulsions is calculated. Furthermore, phase inversion of W/O/W is caused by tearing a thin film of the continuous phase surrounded by large W/O emulsions.

SYMBOLS

C_S	Volume fraction of surfactant in the oil phase (–)
d_{in}	Diameter of internal water droplets (m)
$F_{V,d}$	Volume fraction of the internal water droplets of d_{in} in diameter (–)

H	Height of the center axis of blades from the bottom of vessel (m)
H^*	Normalized height of impeller from the bottom of vessel ($=H/H_T$) (–)
H_T	Height of liquid surface from the bottom of vessel (m)
k	Proportional constant in Equation (3) (m^{-3} s^{-1})
k_0	Constant in Equation (4) (–)
$n_{in,d}$	Number of the internal water droplets of d_{in} in diameter per unit volume of W/O/W emulsion (–)
N	Stirring speed (s^{-1})
t	Stirring time (s)
$v_{in,d}$	Volume of a droplet of d_{in} in diameter (m^3)
v_{in}	Volume of the internal aqueous phase per unit volume of W/O/W emulsion (m^3)

Greek Letters

ϕ_{WO}	Volume fraction of W/O emulsion in W/O/W emulsion (–)

Subscript

0	Beginning of stirring

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