Pressure and temperature effects on the phase transition from a dense droplet to a lamellar structure in a ternary microemulsion

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A small-angle x-ray scattering (SAXS) study of a ternary microemulsion composed of AOT [sodium bis(2-ethylhexyl) sulfosuccinate], water and n-decane was undertaken in order to clarify the phase behavior and the feature of the corresponding structural transition from a dense droplet to a lamellar structure with increasing pressure and temperature. The volume fractions of water and decane were fixed to be equal and the volume fraction of AOT against the whole volume (φ0) was selected to be 0.209 and 0.230 in order to compare results with those obtained by small-angle neutron scattering (SANS). The pressure was varied between 1 and 800 bar under controlled temperature at 20, 25, 29, or 33 °C. Under all conditions applied, the phase transition from the droplet structure to the lamellar structure was observed. The results of analysis of the SAXS profiles indicated that the short-range adhesive potential between droplets becomes more intense with increasing pressure.

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I. INTRODUCTION

The structural features and the basis of self-assembly of microemulsion systems have been intensively investigated. The mixture of AOT, water, and oil [AOT denotes sodium bis(2-ethylhexyl) sulfosuccinate] has been one of the popular systems for such studies. Because the area of the head-group of an AOT molecule is comparably smaller than that of its lipophilic tails, the spontaneous curvature of an AOT membrane is positive (it tends to curve toward water) at ambient temperature and pressure, and a water-in-oil droplet structure is preferable over a large region in the triangle phase diagram, which denotes the fractions of the three components. When the molar ratio of water to AOT is kept constant, for example at 40:8, the droplet radius should be about 50 Å, and only the droplet density φ decreases with increasing oil content. Chen et al. have studied the phase behavior and the structures of these systems from the dilute droplet region to the dense droplet region (0.02 ≤ φ ≤ 0.7)1 with varying temperature and salinity by means of small-angle neutron scattering (SANS), small-angle x-ray scattering (SAXS), static and dynamic light scattering, conductivity measurement, and so on. They showed that the dissociation of the counter-ion from the head-group of AOT molecules is induced with increasing temperature, electrostatic repulsion among head-groups becomes more intense, the spontaneous curvature becomes smaller (a flat membrane is preferable), and the one-phase droplet structure is destabilized. In the dilute droplet region (φ<0.3), the system transforms into a two-phase system in which droplet-rich and droplet-poor phases coexist at high temperature. On the other hand, an ordered lamellar structure appears as a high temperature phase in the dense droplet region. Below these phase boundaries, a percolation threshold exists at φ greater than 0.1.

The effects of pressure on this system have not been as thoroughly studied as those of temperature. In pioneering work on the dilute droplet done by Kim, Bock and Huang, it was shown that increasing pressure induced the same phase separation associated with critical phenomena as increasing temperature.2 Subsequently, Eastoe et al. investigated the effect of pressure on the phase behavior of water/AOT/n-alkane systems by direct visual inspection.3 They showed that both increasing temperature and increasing pressure induced a phase separation in cases where a normal alkane with a carbon number between 6 and 10 was used, i.e., the phase boundary between the single phase and the two-phase regimes was a straight line with the slope dT/dP being negative. On the other hand, the slope had a positive value in cases where they used an alkane with a carbon number below 6. They suggested that the pressure-induced phase separation was dominated by an increase in interdroplet attraction or decreased surfactant-oil compatibility: however, there was no experimental evidence to justify their hypothesis.

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Recently, Nagao and his co-workers have shown that the system composed of AOT and the same volume fraction of water and $n$-decane transformed from a dense droplet structure to a two-phase system with coexistence of lamellar and bicontinuous phases with increasing pressure.\(^4\)\(^-\)\(^9\) They observed SANS profiles from the lower part of the sample and the upper part separately. In the profile from the lower part, a single broad peak at around $Q \sim 0.05 \text{ Å}^{-1}$ which originated from the interdroplet correlation decreased in intensity gradually with increasing pressure, and a quasi-Bragg peak at $Q \sim 0.09 \text{ Å}^{-1}$ corresponding to a regular stacking of membranes started to grow at $P \sim 400 \text{ bar}$. At the same time, little change was observed in the scattering profile in the case of the upper part of the sample; a single broad peak indicative of the droplet structure remained throughout the pressure range examined. They indicated that the characteristic features of the pressure-induced transition were quite similar to those of the temperature-induced transition in terms of the static structure: however, they also suggested that the dynamic features were not the same.\(^8\)\(^,\)\(^10\)

In this study, a comprehensive set of SANS profiles from the AOT water decane system was collected on the $P$-$T$ plane between $1 \leq P \leq 800 \text{ bar}$ and $20^\circ\text{C} \leq T \leq 33^\circ\text{C}$ in order to clarify the effects of pressure and temperature on the phase transition from the water-in-oil droplet to the lamellar structure. The observed SANS profiles of the low pressure and low temperature phase were analyzed within the framework of a polydisperse droplet and a short-range attractive (adhesive) force between droplets. The results indicated that the interdroplet attraction becomes more intense with increasing pressure, as suggested previously by Eastoe et al.\(^3\)

### II. EXPERIMENT

The AOT used in this experiment was purchased from Sigma, and purified by extraction with benzene. Deuterated water of 99% purity obtained from Isotec Inc. was used in order to allow comparison of the results with those obtained by SANS. $N$-decane of 99% purity purchased from Tokyo Kasei Co. Ltd. was used without any treatment. These three ingredients were mixed at two volume fractions of AOT against the whole volume: $\phi_s = 0.209$ and 0.230, with the same volume fraction of water and $n$-decane. The molar ratio of water against AOT was 40.9 for $\phi_s = 0.209$ and 36.2 for $\phi_s = 0.230$.

The SAXS experiments were performed at the BL-15A beam port at the Photon Factory, Institute of Materials Structure Science, High Energy Accelerator Research Organization (KEK). The incident x-ray beam obtained from a bending magnet was monochromatized to be 1.504 Å and its energy resolution ($\Delta E/E$) was about 1%. A one-dimensional proportional counter provided by RIGAKU was placed 1607 mm from the sample position with a vacuum path in between in order to collect the scattered beam up to $Q$ (momentum transfer) = 0.223 Å$^{-1}$. The high-pressure cell used was made of stainless steel with diamond windows. The details of this cell were described in our previous paper.\(^5\) The exposure time for each measurement was 60 sec, and the observed scattering profiles were normalized by the current of the ion chamber placed just before the sample cell in order to compensate for the intensity fluctuation of the beam source. The scattering from the cell was subtracted from all observed data taking the x-ray transmission of the sample into account. The asymmetry of the scattering was checked using an imaging plate as a detector, and all of the observed scattering profiles including those of the high-pressure phase were found to be concentric circles, unlike the case of SANS.\(^6\)\(^,\)\(^7\) Therefore, one-dimensional data were satisfactory to understand the structural features of this system.

### III. RESULTS AND DISCUSSION

Figure 1 shows the changes in the SAXS profiles with increasing pressure for (a) $\phi_s = 0.230$ at $T = 24^\circ\text{C}$ and (b) $\phi_s = 0.230$ at $T = 33^\circ\text{C}$ as a typical example. A single broad peak at $Q \sim 0.05 \text{ Å}^{-1}$ was observed at the ambient pressure. With increasing pressure, the position of the peak shifted to lower-$Q$ gradually, and the peak assimilated to the diffuse scattering centered at $Q = 0$. Above $P_f = 238 \text{ bar}$ for $T = 24^\circ\text{C}$ and $P_f = 82 \text{ bar}$ for $T = 33^\circ\text{C}$, a sharp peak appeared at $Q = 0.08 \text{ Å}^{-1}$, and it grew larger up to $P_f = 726 \text{ bar}$ for $T = 24^\circ\text{C}$ and $P_f = 567 \text{ bar}$ for $T = 33^\circ\text{C}$ shifting toward higher-$Q$. Above $P_f$, little change in the scattering profile was observed. This tendency was almost the same for all of the samples and at all temperatures (excluding the values of $P_f$ and $P_r$), and the behavior was consistent with the results observed by SANS in the case of the lower part of the sample. This is not unexpected, because the x-ray window of the high-pressure cell for SAXS is placed at the lower part of the sample, and one can hardly see the upper part. (See Fig. 1 in Ref. 9.)

The structure of the low-pressure phase is known to be a polydisperse water-in-oil droplet, and it is dominated by a short-range attractive force originating from the hydrophobic interaction between the surfactant tails.\(^11\) Therefore, it is reasonable to use a scattering function of a polydisperse spherically reflecting particle with Schultz size distribution as a form factor $P(Q)$, and a model for a hard sphere with an adhesive surface as a structure factor $S(Q)$. For a polydisperse droplet with a mean radius $R_0$ and a width parameter $Z$ of the Schultz distribution, Kotlarchyk and Chen gave the form factor explicitly as,\(^12\)

$$P(Q) \approx R_0^6(1+Z+1) R_0^6 Z^2 G_1(Q),$$

$$G_1(Q) = \alpha^{-(Z+1)} - (4 + \alpha^2)^{-(Z+1)/\alpha} \cos \left[ (Z+1) \tan \frac{\alpha}{\alpha} \right]$$

$$+ (Z+2)(Z+1) \left[ \alpha^{-(Z+3)} + (4 + \alpha^2)^{-(Z+3)/\alpha} \times \cos \left[ (Z+3) \tan \frac{\alpha}{\alpha} \right] - 2(Z+1)(4 + \alpha^2)^{-(Z+2)/\alpha} \times \sin \left[ (Z+2) \tan \frac{\alpha}{\alpha} \right] \right],$$

$$\alpha = (Z+1)/QR_0,$$
Liu, Chen and Huang introduced a structure factor \( S(Q) \) for particles in the form of a hard sphere with an adhesive surface.\(^{13}\) In their model, the pairwise interparticle interaction potential \( V(r) \) is written as

\[
\frac{V(r)}{k_B T} = \begin{cases} 
+ \infty & \text{for } 0 < r < R' \\
\Omega & \text{for } R' < r < R, \\
0 & \text{for } R < r
\end{cases}
\]

where \( k_B \) is the Boltzmann’s constant, \( T \) the absolute temperature, and \( \Omega \) the attractive potential. The \( R \) means the effective radius of a droplet, which should be the distance between the center of droplet and the corona of the hydrophobic tail of the AOT molecule, and the fractional surface thickness \( \varepsilon = (R - R')/R \) is defined. They included the volume fraction of the droplet \( \phi \), and obtained an analytical form of the structure factor \( S(Q) \) as shown in Eq. (14) in Ref. 13 (which is too complicated to write down here).

Kotlarchyk and Chen\(^{12}\) claimed that the scattering cross section should not be a simple product of \( P(Q) \) and \( S(Q) \) in cases where the droplet size distribution exists or where orientational averaging is necessary, and they introduced the form

\[
d\Sigma \Omega = P(Q)S'(Q),
\]

defining the terms

\[
S'(Q) = 1 + \beta(Q) [S(Q) - 1],
\]

\[
\beta(Q) = |\langle F(Q) \rangle|^2 / |\langle F(Q) \rangle|^2,
\]

where \( \beta(Q) \) is the form factor of the droplet.

FIG. 1. Changes in the SAXS profile with increasing pressure for \( \phi_0 = 0.230 \) at (a) 24 and (b) 33 °C. All profiles are shifted three arbitrary units for better visualization.

| TABLE I. Fit parameters for \( \phi_0 = 0.230 \), \( T = 33 \, ^{\circ} \text{C} \), and \( P = 1 \text{ bar} \). |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| \( \beta(Q) = 1 \) | 51.9±40.4 | 0.28±0.90 | -2.98±19.3 | 0.001±0.001 | 0.001±0.001 | 26.1±7.3 | 40.5±2.8 | 6.78±2.22 | 404±185 | 10.6±3.3 |
| \( \beta(Q) ≠ 1 \) | 53.7±5.6 | 0.27±0.12 | -3.13±49.1 | 0.001±0.005 | 0.001±0.005 | 77.4±1.8 | 40.3±0.5 | 6.66±0.52 | 361±121 | 9.7±2.6 |
where $F(Q)$ is a Fourier transform of the scattering amplitude distribution of a single particle. At the limit of monodispersion, this modification factor $\beta(Q)$ could be approximated to be unity. In our data analysis, the width parameter $Z$ was found to be large (see Table I), i.e., the polydispersity of the system was low, and the values of all of the fit parameters were essentially the same as in the case where $\beta(Q)$ was fixed to be 1 (described below). Therefore, we have considered that the main contribution, which came from the difference in scattering amplitude between the water and oil regions, to the SAXS profile $I(Q)$ could be given by the product of $P(Q)$ and $S(Q)$ assuming $\beta(Q) = 1$ for simplicity.

The scattering amplitude distribution of a droplet in the case of x-ray scattering is shown in Fig. 2. Unlike the case of SANS, the contribution of the thin layers of the head-groups of the surfactant molecules could not be ignored. In this contribution, not only a layer-layer correlation but also a layer-water correlation and a layer-oil correlation should be included. However, the last two correlations should be very small compared with the water-oil correlation and the layer-layer correlation. Thus we introduced only a Lorentzian centered at $Q = 0$ as a representation of surfactant concentration fluctuations, which come from membrane thickness fluctuations and/or crumpling ratio fluctuations. Therefore, in order to explain the SAXS profile of the dense droplet structure at ambient pressure and temperature, the following formula was used:

$$I(Q) = c_0 P(Q) S(Q) + c_2 \frac{\xi_p^{-2}}{Q^2 + \xi_p^{-2}}$$

where $\xi_p$ is the correlation length of the surfactant concentration fluctuations. In this function, the fit parameters are as follows: $R$ the radius of the outer surfactant tails of the droplet, $\phi$ the droplet volume fraction, $\Omega$ the attractive potential, $e$ the fractional surface thickness, $Z$ the width parameter of the Schultz size distribution, $R_0$ the radius of the water core of the droplet, $c_0$ and $c_2$ the coefficients for the water-oil correlation and the layer-layer correlation, respectively, and $\xi_p$. The droplet volume fraction $\phi$ should be conserved in the whole system with increasing pressure; however, its contribution to the observed scattering profile, which came from only the lower part of the sample, could be changed because the system becomes heterogeneous in association with the phase transition. As typical results, the SAXS spectrum in

FIG. 2. Scattering amplitude $\rho$ for a water-in-oil droplet as a function of $r$, the distance from the center of the droplet.

FIG. 3. Observed SAXS profile (open circles) and results of fitting for $\phi_s = 0.230$ at $T = 33^\circ \mathrm{C}$ and at (a) $P = 1$, (b) $P = 200$, and (c) $P = 800$ bar. The solid lines are the curves obtained through fitting to the function described in text. The dashed line in (a) is the result obtained when the modification factor $\beta(Q)$ was not fixed. The inset figure in (a) indicates the structure factors when $\beta(Q)$ was fixed to be unity (solid line), and not fixed (dashed line).
the case where $\phi_s = 0.230$ at $T = 33^\circ C$ and $P = 1$ bar with the fitting result (solid line) is shown in Fig. 3(a), together with the result when $\beta(Q)$ was not fixed to be unity (dashed line). The results show that the fit with $\beta(Q) = 1$ seems to be better than the other. The calculated structure factor $S(Q)$ in the case where $\beta(Q) = 1$ (solid line) and $S'(Q)$ in the case where $\beta(Q) \neq 1$ (dashed line) are depicted in the inset figure of Fig. 3(a), and the values of the fit parameters are given in Table I. These results in the cases where $\beta(Q) = 1$ and $\neq 1$ were essentially the same, and justified the assumption of $\beta(Q) = 1$ described above. Because $Z$ and $\varepsilon$ did not change markedly with increasing pressure, they were fixed throughout the following fitting procedure. In this fitting procedure, seven parameters remained as variables and unique results were obtained for single-peak profiles below $P_s$ [for example, the profiles below 200 bar in Fig. 1(a)].

For the high-pressure lamellar structure, a formula given by Nallet, Roux and Milner was utilized. They separated the scattering into two parts: a quasi-Bragg scattering coming from a regular stacking of the lamellar membranes, and a diffuse scattering corresponding to the surfactant concentration fluctuation. Their function could be simply written as

\begin{table}
\centering
\begin{tabular}{|c|c|c|}
\hline
$T(\degree C)$ & $P_s$(bar) & $P_f$(bar) \\
\hline
\hline
$\phi_s = 0.209$ & \\
\hline
20 & 187 & 569 \\
24 & 127 & 509 \\
29 & 52 & 434 \\
33 & \ldots & 358 \\
\hline
$\phi_s = 0.230$ & \\
\hline
20 & 308 & 796 \\
24 & 238 & 726 \\
29 & 152 & 637 \\
33 & 82 & 567 \\
\hline
\end{tabular}
\caption{Transition pressure.}
\end{table}

FIG. 4. Typical results showing the pressure dependence of two times the corona radius $R$ (open circles) and the characteristic repeat distance $d_z$ (full triangles) of the lamellar structure for $\phi_s = 0.230$ at $T = 33^\circ C$. The transition start pressure $P_s$ and the finish pressure $P_f$ could be defined from this plot.

FIG. 5. The $P-T$ phase diagram obtained. Both the slope in the case of $P_s$ and that in the case of $P_f$ were almost the same negative value.

FIG. 6. Summary of the pressure dependence of (a) $2R_z$ and (b) $R_0$ for $\phi_s = 0.230$ as a function of $P - P_s$. It is clear that both $R$ and $R_0$ depended on the temperature and that the pressure dependence of $R$ and $R_0$ could be normalized as a function of $P - P_s$. 

TABLE II. Transition pressure.
where $d_L$ and $\xi$ indicate the mean repeat distance of the lamellar membranes and its correlation length, respectively. In our data, the second order Bragg reflection was observed at $Q = \frac{4 \pi}{d_L}$, and a term,

$$I(Q) = c_1 \frac{\xi^{-2}}{(Q - \frac{2 \pi}{d_L})^2 + \xi^{-2}} + c_2 \frac{\xi_\rho^{-2}}{Q^2 + \xi_\rho^{-2}}, \quad (9)$$

was necessary, where $c_1'$ is the coefficient of this term, and $\xi'$ is the correlation length of membranes defined from the second order reflection. However, the contribution of this term was relatively small and the fit results for $c_1'$ and $\xi'$ scattered randomly, the ratios $c_1'/c_1$ and $\xi'/\xi$ were fixed to be 0.146 and 0.5, respectively. Because the second terms in Eqs. (8) and (9) are the same, the equation

$$I(Q) = c_0P(Q)S(Q) + c_1 \frac{\xi^{-2}}{(Q - \frac{2 \pi}{d_L})^2 + \xi^{-2}} + c_2 \frac{\xi_\rho^{-2}}{Q^2 + \xi_\rho^{-2}}, \quad (10)$$

could be used for the transient state between $P_s$ and $P_f$, where the low-pressure dense droplet structure and the lamellar structure coexist. In this function, nine parameters remained as variables and unique and relevant results were obtained for fitting to double peak profiles [for example, the profiles between 300 bar and 600 bar in Fig. 1(a)]. On the other hand, it was not easy to fit the function (10) to single peak profiles just below $P_f$ [for example, the profile at 700 bar in Fig. 1(a)] because the first term merged into the third. Therefore, just below $P_f$, the errors of $R$, $\phi$, $\Omega$, $R_0$, $c_0$, $c_2$, and $\xi_\rho$ were relatively large. Typical results of fitting are shown in Figs. 3(b) ($\phi_s = 0.230$, $T = 33 \degree C$, $P = 200$ bar: the transient state) and 3(c) ($\phi_s = 0.230$, $T = 33 \degree C$, $P = 800$ bar: the lamellar phase).

In Fig. 4, the pressure dependence of $2R$ and $d_L$ for $\phi_s = 0.230$, $T = 33 \degree C$ is shown as a function of pressure on
the same plot. $2R$, which is twice the effective radius of a droplet, should have a close relation to the mean droplet-droplet distance, which might correspond to the characteristic repeat distance $d_m$ obtained by SANS$^6$ using the model proposed by Teubner and Strey.$^{15}$ Therefore, the similarity of this figure with Fig. 5 in Ref. 6 might justify the present data analysis. The transition start pressure $P_f$ could be defined where the peak indicative of the lamellar structure appeared, and the transition finish pressure $P_f$ where the increase in $2R$ stopped (or the contribution of the low-pressure phase vanished). Observed values of $P_f$ and $P_f$ for various conditions are summarized in Table II and plotted in Fig. 5. The phase boundaries on the $P-T$ plane could be approximated as straight lines, which are consistent with the lines defined by Eastoe et al.$^3$ It is to be noted that the difference between $P_f$ and $P_f$ did not depend on temperature; $P_f-P_f=382$ bar for $\phi_s=0.209$ and $P_f-P_f=486$ bar for $\phi_s=0.230$.

In Fig. 6, the pressure dependence of (a) $2R$ and $d_L$ and (b) $R_0$ for $\phi_s=0.230$ at all temperatures examined is summarized as a function of $P-P_s$. It is clear that the value of $R$ and that of $R_0$ decreased slightly with increasing temperature: however, the tendency of the pressure dependence is the same: $R$ decreased below $P-P_s\approx 150$ bar and increased drastically up to $P=P_f$, and remained constant above $P_f$. The $d_L$ decreased slightly with increasing pressure, but its temperature dependence was not remarkable. The water core radius $R_0$ depended on temperature: however, no change was observed with increasing pressure. (Note that the decrease in $R_0$ above $P=P_f$ should have no meaning, because the contribution of droplet structure in this regime to the SAXS profile was very weak and one could not define this parameter precisely.) The results for $\phi_s=0.209$ were almost the same as for $\phi_s=0.230$. These observations served to verify the difference in the effects of temperature and pressure on the droplet structure; temperature makes the water-core radius smaller, and pressure changes the effective size of the corona of the hydrocarbon tail of the AOT molecules.

Figure 7 summarizes the pressure dependence of (a) the surface adhesion potential $\Omega$, (b) the droplet density $\phi$, (c) the correlation length of the lamellar membrane $\xi$ and (d) the correlation length of the surfactant concentration $\xi_p$. Because the error of $\Omega$ was very large (see Table I), the values themselves may be of little importance. However, the values around $-3k_BT$ are reasonable and the tendency that the potential increased in its depth with increasing pressure is clear. Therefore, we can conclude that this result is evidence that the interdroplet attractive force becomes intense with increasing pressure and this could be the origin of the pressure-induced phase transition, unlike the temperature-induced transition. It is not yet clear how to interpret the decrease in $\phi$ with increasing pressure: however, it has a direct relation with the number density and the droplet radius. In this phase transition process, deformation, incorporation, and dissociation of droplets may occur. Therefore, an apparent decrease in $\phi$ was observed. We did not find any pressure or temperature dependencies of $\xi$ and $\xi_p$, and they might be constant.

IV. CONCLUSION

In this study, the temperature and pressure dependence of a ternary microemulsion were investigated at $20^\circ C\leq T \leq 33^\circ C$ and $1\text{ bar} \leq P \leq 800$ bar. The SAXS profiles observed in the low-pressure phase were explained using a model describing polydisperse droplets in the form of a hard sphere and an adhesive interdroplet interaction. The transition start pressure $P_s$ and the transition finish pressure $P_f$ decreased with increasing temperature, and the phase boundaries could be approximated by straight lines with the slope $dP/dT=\approx -17.5$ bar/°C independently of $\phi_s$, i.e., the droplet size. This result is inconsistent with the result of Eastoe et al.,$^3$ who showed that the transition temperature is more pressure sensitive for smaller droplets. However, our AOT concentrations measured were only two points, and it should be investigated more carefully in the future. With increasing pressure, the effective size of the corona (the hydrocarbon tails of AOT) and the depth of the attractive potential increased, whereas the size of the water core remained unchanged. These results clearly indicated that the structure and the interactions of the surfactant tail are predominant factors affecting the pressure-induced phase transition.

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