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Model calculations of aggregation numbers and radii of w/o microemulsions

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With 4 figures and 3 tables

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1. Introduction

The concept microemulsions describing transparent solutions consisting of water/oil/alcohol (often called "co-surfactant")/surfactant was first introduced by *J. H. Schulman,* as a result of comprehensive studies (1–4). Ever since then, there has been much discussion whether ternary water/co-surfactant/surfactant mixtures built up by reversed micellar aggregates can be classified as w/o microemulsions. Since the thermodynamic stability is a fundamental criterion, the question of the size and dimensions of the appearing aggregates can be of some importance in distinguishing between these denominations (5). If, however, completely apolar additives are dissolved in the intermicellar solution and have only a negligible effect on the size of the aqueous core of the reversed micelle, it is obvious that the micellar solution must be identified with a w/o microemulsion. It is our opinion that the alcohol rich, homogeneous solution phases (L_2 phases) investigated by *Ekwall* et al. (6–9) and *Danielsson* et al. (10–12) have the properties of a w/o microemulsion, a fact also emphasized by other authors (13–15). It must, however, be pointed out that the L_2 phase is only one part of the complicated phase equilibria occurring in ternary phase diagrams, as illustrated in figure 1. In our model system (fig. 1) consisting of water (H_2O)/decanol ($C_{10}OH$)/sodium octanoate (NaC_8) (8, 10) the

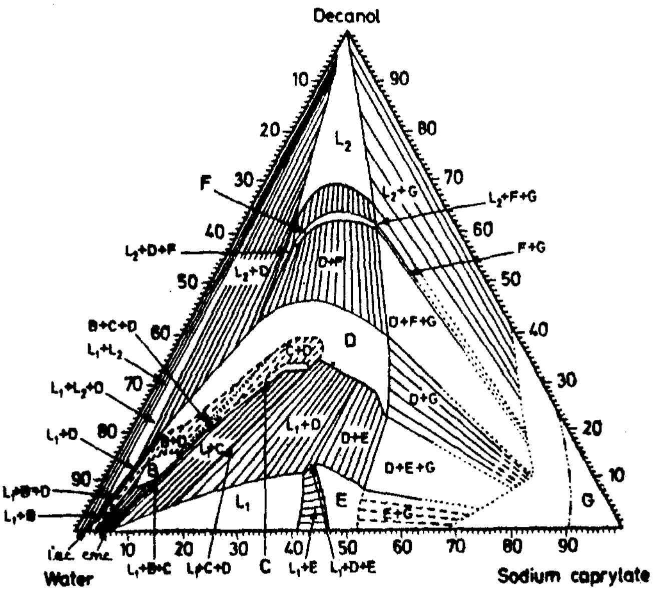

Fig. 1. Phase diagram for the three-component system water/decanol/sodium octanoate at 293 K. Concentrations are expressed as per cent by weight (6–12)

present work is restricted to the phases denoted by F and L_2. The former is a mesophase built up by hexagonally ordered rod-shaped aggregates with a polar aqueous interior, while the lipophilic moiety of the surfactant is oriented towards the surrounding medium (8, 10, 16). The L_2 phase, as already mentioned, is a solution phase consisting of reversed micelles with a constitution resembling of the described mesoaggregate, but probably with a different geometrical shape, i. e. a spherical form (6, 7, 9).

The usefulness of estimating aggregation numbers and radii of micelles is evident. It is our aim to derive a formula, based upon X-ray findings and density data, for this purpose. As a first part we here present a method for performing these calculations on reversed micelles. We will later extend the derivation to micelles in an aqueous medium.

2. Derivation of formula

The purpose of the derivation is to calculate aggregation numbers and radii of the reversed micelles in the L_2 phase (fig. 1). We start from the F phase, the structure of which is known from X-ray studies and calculate the total area occupied by each carboxylic group at the interface water/hydrocarbon. By choosing an appropriate surface element (fig. 2) with the thickness $\sqrt{A}$, where A is the area per carboxylic group, we obtain a prism-shaped volume element. We denote water, the polar endgroup of the surfactant and its counterion by the index s (solubilized state) and obtain according to figure 2

where N_{agg} is the aggregation number. By using the value of A from formula [5], equations [8] and [9] supply the radius and the aggregation number of the reversed micelle. It must, however, once more be stressed that r_s denotes the radius of the solubilized region and thus does not include the lipophilic moieties.

$$m_s = \varrho_s \cdot \frac{1}{2} \cdot \pi \cdot r_s^2 \cdot \sqrt{A} \qquad [1]$$

where m_s is the mass of the solubilized species, ϱ_s is the density and r_s the radius. The total mass of the volume element,

$$m_{tot} = \varrho_{tot} \cdot \frac{d^2}{\sqrt{3}} \cdot \sqrt{A} \qquad [2]$$

where ϱ_{tot} is the density of the solution and d is the Bragg spacing according to the most intense line. By combining [1] and [2] the weight fraction of the solubilized state, w_s, can be expressed by

$$w_s = \frac{m_s}{m_{tot}} = \frac{\sqrt{3} \cdot \pi}{2} \cdot \left(\frac{\varrho_s}{\varrho_{tot}}\right) \cdot \left(\frac{r_s}{d}\right)^2 . \qquad [3]$$

Approximating $\varrho_s = \varrho_{H_2O}$, the density of pure water, formula [3] gives the value of r_s. Since the composition of the solution is known, it is possible to determine w_s from

$$w_s = \left(\frac{n_{C_{10}OH}}{n_{NaC_8}}\right)_i \cdot M_{OH} \frac{w_{NaC_8}}{M_{NaC_8}} + \frac{M_{COONa}}{M_{NaC_8}} \cdot w_{NaC_8} + w_{H_2O} \qquad [4]$$

where $(n_{C_{10}OH}/n_{NaC_8})_i$ is the molar ratio of cosurfactant to surfactant at the interface water/hydrocarbon, M_j is the molar mass of component j, while w_j is its weight fraction. When calculating the total area A one must notice that the weight fraction of sodium octanoate, w_{NaC_8}, in figure 2 is given by the expression

$$w_{NaC_8} = \left(\frac{\pi \cdot r_s \cdot M_{NaC_8}}{\sqrt{A} \cdot N_A}\right) \Big/ \left(\frac{\sqrt{A} \cdot d^2 \cdot \varrho_{tot}}{\sqrt{3}}\right) \qquad [5]$$

where N_A is Avogadro constant. Formula [5] supplies the value of A from the F phase.

Since there do not exist so highly ordered structures and strict geometries in the L_2 phase as in the F phase, it is impossible to develop a similar derivation for that phase. Therefore we observe that

$$V_{tot} = V_m + V_s \qquad [6]$$

where V_{tot} is the volume of the solution, V_m and V_s is the volume of the medium and the solubilized state, i. e. the micellar core, respectively. V_{tot} is determined from density data. For V_m we have

$$V_m = \frac{(w_{C_{10}OH})_m}{\varrho_{C_{10}OH}} + \frac{M_{COONa} \cdot w_{NaC_8}}{M_{NaC_8} \cdot \varrho_{C_7H_{16}}} + \frac{(M_{C_{10}OH} - M_{OH}) \cdot (w_{C_{10}OH})_s}{M_{C_{10}OH} \cdot \varrho_{C_{10}H_{22}}} + \frac{(w_{H_2O})_m}{\varrho_{H_2O}} \qquad [7]$$

where ϱ_j denotes the density of component j and the lower indexes m and s are defined above. By introducing a spherical shape as an approximation for the micellar form, we obtain

$$V_s = \frac{w_{NaC_8} \cdot N_A}{M_{NaC_8} \cdot N_{agg}} \cdot \frac{4\pi}{3} \cdot r_s^3 \qquad [8]$$

and further

$$4\pi r_s^2 = N_{agg} \cdot A \qquad [9]$$

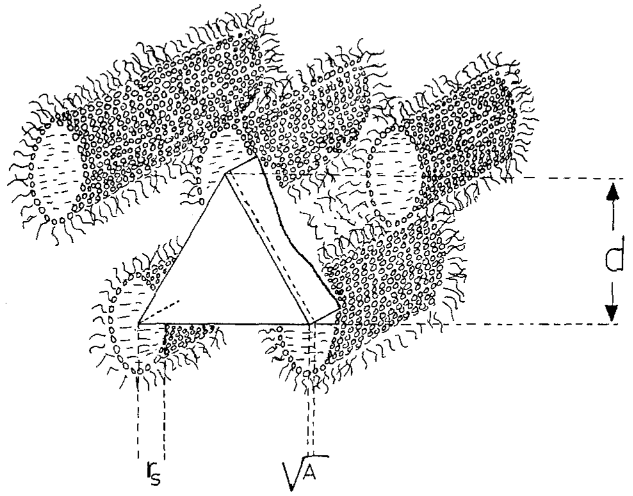

Fig. 2. Schematic illustration of the prism-shaped volume element in the reversed hexagonal mesophase F

3. Numerical calculations

In the absence of own experimental data at 298 K, we have used data, published earlier by *Ekwall* et al. (6, 16, 17) at 293 K. Some calculations for the F phase are summarized in table 1. The high values of the area A indicate that the total area cannot be attributed to the carboxylic group alone. Thus these data take into account the position of decanol molecules at the interface water/hydrocarbon. Since, however, the exact number of hydroxylic groups in this position is not available, table 1 shows the influence of a varying ratio $(n_{C_{10}OH}/n_{NaC_8})_i$ on the values of r_s and A. According to table 1 and earlier published data (18) the most probable value of this ratio is 2 in the waterpoor and 4–5 in the water-rich parts of the F phase. In order to give a more complete picture of the influence, we have included in the table the whole range of $(n_{C_{10}OH}/n_{NaC_8})_i$ with the maximum value given by the composition.

Table 1. The radii r_s and areas A for mesoaggregates in the F phase. The values are calculated from formula [3], [4] and [5] for different ratios $(n_{C_{10}OH}/n_{NaC_8})_i$ at the interface water/hydrocarbon

w_{NaC_8}	$w_{C_{10}OH}$	w_{H_2O}	$\frac{n_{H_2O}}{n_{NaC_8}}$	$\frac{n_{C_{10}OH}}{n_{NaC_8}}$	$(n_{C_{10}OH}/n_{NaC_8})_i = 0$		$(n_{C_{10}OH}/n_{NaC_8})_i = 1$		$(n_{C_{10}OH}/n_{NaC_8})_i = 2$		$(n_{C_{10}OH}/n_{NaC_8})_i = 3$		$(n_{C_{10}OH}/n_{NaC_8})_i = 4$		$(n_{C_{10}OH}/n_{NaC_8})_i = 5$	
					r_s/nm	A/nm^2	r_s/nm	A/nm^2	r_s/nm	A/nm^2	r_s/nm	A/nm^2	r_s/nm	A/nm^2	r_s/nm	A/nm^2
0.2337	0.6149	0.1514	5.98	2.76	0.78	0.74	0.82	0.78	0.85	0.81						
0.1800	0.6301	0.1899	9.74	3.68	0.88	0.92	0.91	0.95	0.94	0.98	0.96	1.01				
0.1502	0.6237	0.2261	13.89	4.36	1.00	1.05	1.03	1.08	1.06	1.11	1.08	1.13	1.11	1.16		
0.1303	0.6228	0.2469	17.49	5.02	1.08	1.18	1.10	1.21	1.12	1.23	1.15	1.26	1.17	1.28	1.19	1.31
0.1281	0.6119	0.2600	18.73	5.02	1.13	1.20	1.15	1.22	1.17	1.24	1.19	1.27	1.22	1.29	1.24	1.31
0.1235	0.5905	0.2860	21.37	5.02	1.25	1.20	1.28	1.22	1.30	1.24	1.32	1.26	1.34	1.29	1.37	1.31

Table 2. The radii r_s and the aggregation numbers N_{agg} of reversed micelles with all water in the micellar core, i. e. $(n_{H_2O}/n_{C_{10}OH})_m = 0$, and with a decanolic medium saturated with water, i. e. $(n_{H_2O}/n_{C_{10}OH})_m = 0.356$, respectively

					$(n_{H_2O}/n_{C_{10}OH})_m = 0$		$(n_{H_2O}/n_{C_{10}OH})_m = 0.356$	
w_{NaC_8}	$w_{C_{10}OH}$	w_{H_2O}	$\frac{n_{H_2O}}{n_{NaC_8}}$	$\frac{n_{C_{10}OH}}{n_{NaC_8}}$	r_s/nm	N_{agg}	r_s/nm	N_{agg}
0.1734	0.7010	0.1256	6.65	4.25	0.70	7.4	0.53	4.3
0.1469	0.6999	0.1532	9.62	5.00	0.90	11.1	0.73	7.2
0.1266	0.6954	0.1780	12.98	5.77	1.14	16.6	0.95	11.6
0.0957	0.7575	0.1468	14.15	8.31	1.12	14.7	0.87	8.9
0.1021	0.6899	0.2080	18.79	7.10	1.35	19.2	1.16	14.2

Table 2 illustrates the situation in the L_2 phase. Here we have to consider a distribution of water molecules between the solubilized state and the decanolic medium, as previously pointed out (19). In the absence of vapour pressure data we only deal with the alternatives of a water-free medium, i. e.

$(n_{H_2O}/n_{C_{10}OH})_m = 0$, and a saturated medium, i. e. $(n_{H_2O}/n_{C_{10}OH})_m = 0.356$, respectively. It must be noticed that table 2 does not include decanol in the micelle and must thus be regarded a first approximation. When considering an interface built up by as well sodium octanoate as decanol molecules the weight fractions in equation [7] change and other values are obtained. Table 3 illustrates, however, these aspects. An estimation of the water content in the medium as a function of a varying interfacial ratio $(n_{C_{10}OH}/n_{NaC_8})_i$ is given in figure 3. The figure is based upon a saturation of the decanolic medium. Instead of performing the calculations for different ratios (n_{H_2O}/n_{NaC_8}), valuable information is obtained by keeping this ratio constant and by varying the content of decanol. Figure 4 shows the variations in r_s along $n_{H_2O}/n_{NaC_8} = 18.79$ as a function of the decanol content expressed as the weight fraction.

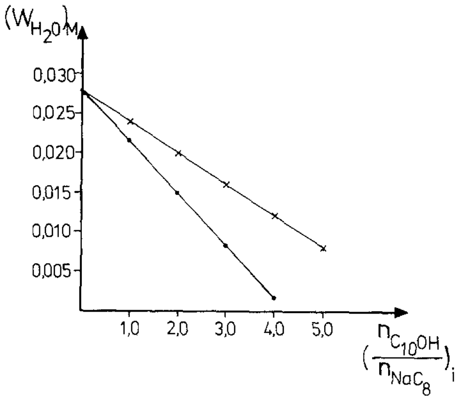

Fig. 3. The calculated weight fraction of water in the decanolic medium as a function of the molar ratio decanol/sodium octanoate at the interface water/hydrocarbon in the L_2 phase. The basis of the calculation is a saturation of the decanolic medium with water. × represents $n_{H_2O}/n_{NaC_8} = 18.79$ and ● $n_{H_2O}/n_{NaC_8} = 6.65$, respectively

4. Discussion

4.1 The hexagonal mesophase F

A comparison between the radii in table 1 and data published earlier (18) display a slight discrepancy. It is, however, to be kept in mind that different methods of calculating the values give quantities r with another meaning. Our r_s represent only the extension of the polar interior of the mesoaggregate thus completely excluding

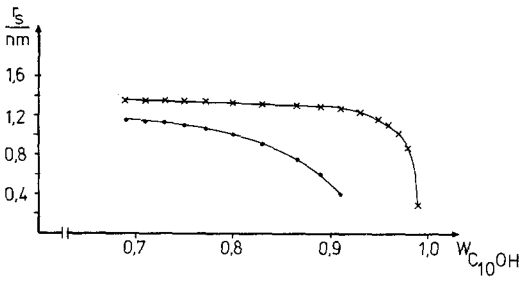

Fig. 4. The calculated extension of the aqueous core of the reversed micelle in the L_2 phase versus the weight fraction of decanol for $n_{H_2O}/n_{NaC_8} = 18.79$. × represents $(n_{H_2O}/n_{C_{10}OH})_m = 0$ and ● $(n_{H_2O}/n_{C_{10}OH})_m = 0.356$, respectively

the lipophilic moieties of the surfactant and the co-surfactant. The order of magnitude of the calculated surface values requires a special comment. Since A reaches a value of 0.8–1.30 nm^2, it is of course impossible for a single carboxylic group to cover such an area alone. Thus A includes a number of hydroxylic groups. If the COO^- group is assigned a surface of 0.30–0.35 nm^2 and the OH group 0.22–0.28 nm^2 (20–22) the total area A is accordingly composed of approximately 4–5 decanol/sodium octanoate in the water-rich and 2 decanol/sodium octanoate in the water-poor part of the mesophase F. From these calculations it can be concluded that the major part of the decanol molecules participate in stabilizing the formed mesoaggregates. Since the content of decanol, considered a medium, is extremely low we do not have to take into account any distribution of water molecules between different states, as in the L_2 phase.

4.2 The solution phase L_2

In order to carry out the calculations of the aggregation numbers and radii of the reversed micelles in the L_2 phase, it is necessary to make use of the equilibrium with the mesophase F. By putting the same surface charge density forward as a condition on this equilibrium, it is possible to conclude the characterization of the micelles. The mathematical interpretation of the expressed criterion is that A in equations [5] and [9]) obtains the same value. One must notice, however, that strictly speaking the condition is fulfilled only along a tieline crossing the two-phase region $L_2 + F$ (fig. 1). If we extend the validity of the condition to include the inner parts of the L_2 phase, the presentation of figure 4 is justified.

4.2.1 An approximately constant content of decanol and a varying ratio n_{H_2O}/n_{NaC_8}

Tables 2 and 3 illustrate the decreasing trend in as well the micellar core as the aggregation number when proceeding from a higher ratio of n_{H_2O}/n_{NaC_8} to a lower. The parallel behaviour in r_s from the F phase is conspicuous. Table 2 shows the influence of a saturation of the decanolic medium with water. In r_s this is reflected as a decrease of about 0.2 nm. When making the corrections for the participating decanol molecules at the interface in formula [7],

Table 3. The radii r_s and aggregation numbers N_{agg} for reversed micelles assuming a spherical shape for the aggregate and a saturation of the decanolic medium with water, i. e. $(n_{H_2O}/n_{C_{10}OH})_m = 0.356$, for different ratios $(n_{C_{10}OH}/n_{NaC_8})_i$ at the interface water/hydrocarbon

					$(n_{C_{10}OH}/n_{NaC_8})_i = 0$		$(n_{C_{10}OH}/n_{NaC_8})_i = 1$		$(n_{C_{10}OH}/n_{NaC_8})_i = 2$		$(n_{C_{10}OH}/n_{NaC_8})_i = 3$		$(n_{C_{10}OH}/n_{NaC_8})_i = 4$		$(n_{C_{10}OH}/n_{NaC_8})_i = 5$	
w_{NaC_8}	$w_{C_{10}OH}$	w_{H_2O}	$\frac{n_{H_2O}}{n_{NaC_8}}$	$\frac{n_{C_{10}OH}}{n_{NaC_8}}$	r_s/nm	N_{agg}	r_s/nm	N_{agg}	r_s/nm	N_{agg}	r_s/nm	N_{agg}	r_s/nm	N_{agg}	r_s/nm	N_{agg}
0.1734	0.7010	0.1256	6.65	4.25	0.53	4.3	0.54	4.2	0.54	4.1	0.55	4.0	0.55	3.9		
0.1469	0.6999	0.1532	9.62	5.00	0.73	7.2	0.73	6.9	0.72	6.7	0.72	6.5	0.73	6.3	0.73	6.2
0.1266	0.6954	0.1780	12.98	5.77	0.95	11.6	0.95	11.2	0.94	10.7	0.94	10.4	0.93	10.0	0.93	9.6
0.0957	0.7575	0.1468	14.15	8.31	0.87	8.9	0.87	8.7	0.87	8.5	0.87	8.2	0.87	8.0	0.87	7.8
0.1021	0.6899	0.2080	18.79	7.10	1.16	14.2	1.16	13.8	1.15	13.4	1.15	13.0	1.14	12.7	1.14	12.4

the decrease is reduced to about 0.1 nm due to a corresponding increase in the total area, A, by approximately 0.03 nm^2. Quasielastic light-scattering measurements (23) give for the solution with a radius of 0.87 nm in table 3, a Stokes radius of 1.4 nm. These values are in agreement with each other, since the experimental result characterizes the whole kinetic unit, thus including the lipophilic moieties. The data reported by other authors (5, 15) agree with our theoretical results, although the former concern other systems.

From the aggregation numbers, it is possible to estimate the molar mass, M_m, of the reversed aggregate. The result in the tables renders a M_m value of 4.5 kg mol^{-1} in the water-poor part, i. e. for $n_{H_2O}/n_{NaC_8} = 6{,}65$ and 21.8 kg mol^{-1} in the water-rich part with a molar ratio of 18.79. When we, however, correct for the water bond to the decanol in the medium, the corresponding values are 2.4 and 13.8 kg mol^{-1}, respectively. Ultracentrifugations (24) of a solution with a molar ratio of water to sodium octanoate = 22.5 and 67 weight per cent decanol rendered a molar mass of 15.6 kg mol^{-1}, which agrees well with 13.8 kg mol^{-1} for $n_{H_2O}/n_{NaC_8} = 18.79$ above.

4.2.2 A constant ratio n_{H_2O}/n_{NaC_8} and a varying content of decanol

This situation for a molar ratio of 18.79 is described in figure 4. The upper curve based upon the rough calculation in table 2 with $(n_{H_2O}/n_{NaC_8})_m = 0$, permit an almost constant radius uptil about 94 weight per cent decanol, whereafter the model obviously collapses. The continuously decreasing lower curve reflect the situation with a less water content in the micellar core. It is evident that the latter curve support the idea of a "step-wise" association with a pronounced difficulty of observing a distinctly defined critical micelle concentration (c.m.c.) in the reversed micellar solutions (5, 15, 25–27).

5. Concluding remarks

It is well-known that theoretical estimations of radii and aggregation numbers of micellar aggregates are connected with several difficulties. By making use of the equilibria between mesophases, the structure of which are known basically from X-ray findings valuably complemented by NMR- and dielectric studies (27–33), and solution phases, we find it possible to proceed with the calculations. Expressing the equilibrium in terms of an equal surface charge density between mesoaggregates and micelles, one obtains the mathematical basis for the derivation of the wanted quantities.

In this report we have presented calculations on some w/o microemulsions at 293 K. The obtained values agree well with experimental data. Extensive X-ray and density measurements at 298 K are already initiated in our model system consisting of water/decanol/sodium octanoate with the aim of a comprehensive characterization of the micellar aggregates in the both solution phases L_1 and L_2.

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Summary

A method of calculating aggregation numbers and radii of reversed micellar aggregates is presented and applicated on the alcohol-rich solution phase L_2 in the model system water/decanol/sodium octanoate at 293 K. The result showing good agreement with experimental data illustrate the extension of the micellar core at different decanol contents and molar ratios water to sodium octanoate. The influence of a distribution of water between the interior of the micelle and the decanolic medium is demonstrated.

Zusammenfassung

Es wird eine Methode zur Berechnung von Aggregationszahlen und Radien invertierter mizellarer Aggregate angegeben und auf die Alkohol-reichen L_2-Lösungen im Modellsystem Wasser/Dekanol/Natrium-Oktanoat bei 293 K angewendet. Die Resultate sind in guter Übereinstimmung mit experimentellen Messungen; sie zeigen die Ausdehnung der Mizellkerne bei verschiedenen Dekanol-Konzentrationen und bei verschiedenen molaren Verhältnissen Wasser/Oktanoat. Der Einfluß der Verteilung von Wasser zwischen dem dekanolischen Medium und den Mizellen wird diskutiert.

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