

01 Jan 1979

A Simple Experiment Illustrating The Structure Of Association Colloids

Stig Friberg

Missouri University of Science and Technology, stic30kan@gmail.com

Beverly Bendiksen

Follow this and additional works at: https://scholarsmine.mst.edu/chem_facwork

 Part of the [Chemistry Commons](#)

Recommended Citation

S. Friberg and B. Bendiksen, "A Simple Experiment Illustrating The Structure Of Association Colloids," *Journal of Chemical Education*, vol. 56, no. 8, pp. 553 - 555, American Chemical Society, Jan 1979.
The definitive version is available at <https://doi.org/10.1021/ed056p553>

This Article - Journal is brought to you for free and open access by Scholars' Mine. It has been accepted for inclusion in Chemistry Faculty Research & Creative Works by an authorized administrator of Scholars' Mine. This work is protected by U. S. Copyright Law. Unauthorized use including reproduction for redistribution requires the permission of the copyright holder. For more information, please contact scholarsmine@mst.edu.

A Simple Experiment Illustrating the Structure of Association Colloids

The energy crisis and the predicted petroleum shortage have recently focused attention on the structure of association colloids, such as normal and inverse micellar solutions and the different lyotropic liquid crystals found in surfactant systems. Such association structures form the basis of microemulsions (1-7), which are the most promising agents in tertiary oil recovery (8) and also offer a means of reduction of the harmful nitrous oxides in the exhaust gases from diesel engines (9). Lyotropic liquid crystals, especially the lamellar type (10) are good models for biological membranes (11) and other interesting biological phenomena involving order. The recent observations (of the disordering of the intercellular matrix as a preliminary step in the formation of cancer cells) are extremely interesting (12).

Unfortunately, little or no information on such systems is given in most physical chemistry curricula and useful experiments for demonstrating association colloids are virtually non-existent.

In this article we describe a simple experiment that illustrates the intermolecular phenomena involved in three association structures, normal and inverse micelles and lyotropic liquid crystals. The concepts of solubilization, microemulsion, and emulsion are introduced.

The Experiment (3 hours)

Make two identical surfactant solutions by dissolving 2.50 g of sodium dodecyl sulfate (Gallard-Schlesinger, Specially pure) in 10.0 ml distilled water at room temperature. (The solution may be heated in a hot water bath to facilitate dissolution). Excessive shaking should be avoided, to prevent foaming. To one solution, add 18.75 g of pentanol (Fischer, Practical grade) in the following portions (Series I).

Addition	Amount, g	Total Added, g
1	0	0
2	0.100	0.100
3	0.900	1.000
4	2.250	3.250
5	5.250	8.500
6	10.250	18.750

To the other solution, add octane (Eastman, Practical grade) in identical amounts.

Observe the behavior of the solutions (transparency, transmission of polarized light between crossed polarizing films, and electrical conductance) after each addition.

After the final addition of alcohol to Series I, add 20.0 g of the hydrocarbon (octane).

The Results

Series I—Addition of alcohol to the aqueous solution

The first two additions of pentanol gives samples (Nos. 2, 3) that are optically isotropic; they appear black between crossed polarizing films.¹ The electrical conductance [Samples No. (1) 69,200, (2) 69,200 and (3) 62,500 μ mhos] are of the same magnitude as that of the original aqueous solution.

Sample No. 4 is viscous, radiates light when placed between the crossed polarizers, gives a characteristic pattern in the microscope with polarized light (Fig. 1), and is not suitable for the determination of electrical conductivity. Samples No. 5 and No. 6 are similar to Samples No. 1 through No. 3 except that their electrical conductivity is much lower (8000, 1000 μ mhos).

¹ Observe the sample *against* a desk lamp between two polarizing films (Bausch & Lomb) that have been arranged to extinguish the maximum amount of light.

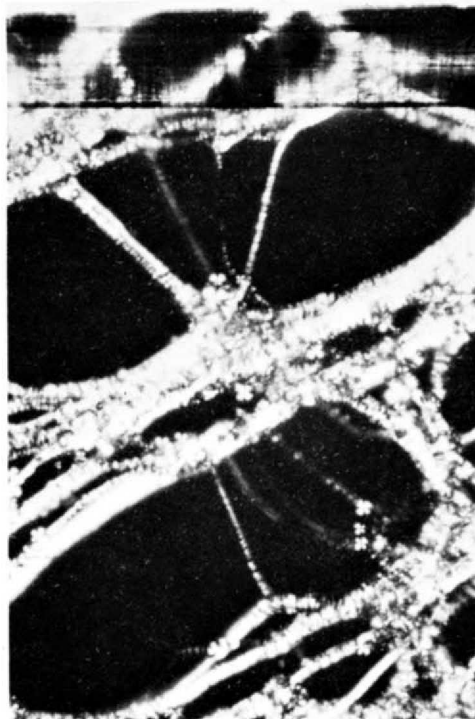


Figure 1. The anisotropic nature of the liquid crystalline phase is demonstrated by its microscopy pattern in polarized light.

Series II—Addition of hydrocarbon to the aqueous solution

Sample No. 2 is identical to the one with alcohol, but the remaining samples will be unstable, turbid emulsions, unsuitable for conductivity measurement. The individual droplets of the emulsions may be observed in a microscope.

Addition of hydrocarbon to the final alcohol sample

The hydrocarbon will dissolve completely leaving an isotropic solution of low conductivity.

Explanation of the Results

The results demonstrate the importance of the presence and structure of the polar group for the intermolecular forces giving rise to association structures. The sodium dodecyl sulfate and pentanol are examples of amphiphilic structures; they both consist of a non-polar hydrocarbon chain and a polar group (Fig. 2).

Sample No. 1, the solution of sodium dodecyl sulfate in water is a normal micellar solution (Fig. 3A), since the amount of the surfactant present in solution far exceeds the critical micellization concentration for $C_{12}H_{25}OSO_3Na/H_2O$. To define this further then, for each system of surfactant/water there exists a specific concentration of that substance at which micelles begin to form. This process can be described as follows.

When an amphiphilic substance such as sodium dodecyl sulfate is dissolved in water it adsorbs preferentially to the surface of the solution (Fig. 4). The sparingly soluble non-polar parts extend from the surface toward the air, their presence weakening the intermolecular forces at the air/water interface and the surface free energy (= surface tension for a liquid) is reduced. This reduction of surface tension with increasing surfactant concentration (Fig. 4) ceases in a certain small concentration range. The surface is now saturated with surfactant. Further additions do not lead to an increase of the concentration of surfactants at the surface. Instead, the surfactant forms a new interface in the solution by an aggregation to normal micelles (Fig. 3A);

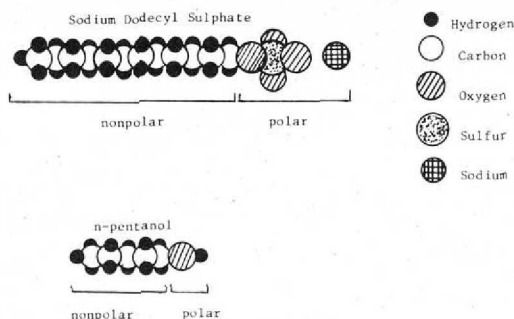


Figure 2. Amphiphilic substances consist of a non-polar (hydrophobic) part and a polar (hydrophilic) part.

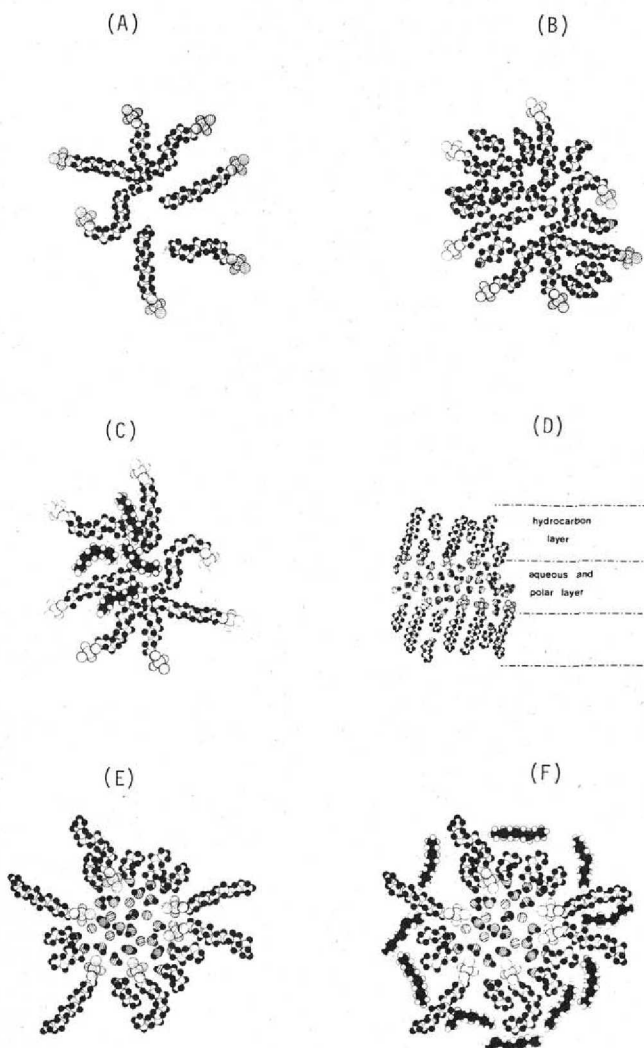


Figure 3. The normal micelle (A) may solubilize hydrophobic amphiphiles such as the pentanol (B) or hydrocarbons (C). At enhanced level of pentanol a phase separation takes place to a lamellar liquid crystal (D); further increase of the pentanol concentration gives rise to an alcohol solution with inverse micelles (E). In this solution large amounts of hydrocarbon (F) may be dissolved to a microemulsion.

these isolate the non-polar parts of the molecule from the aqueous solution by the interface formed from the polar layer orienting towards the water. The micelles contain on the order of 25 surfactant molecules each and have a radius of approximately 30 Å. Most of the sodium ions (~85%) are attached to the micelle and the remaining ones are free in the aqueous solution giving rise to the conductivity of Solutions No. 1 through 3.

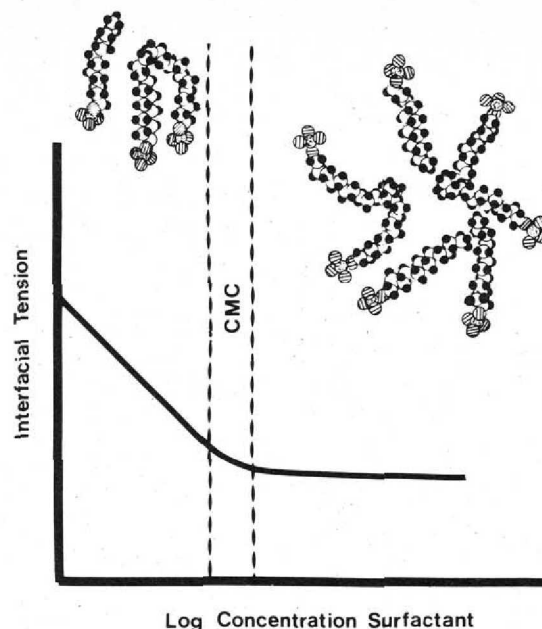


Figure 4. Surfactants adsorb strongly to a water/air or water/oil interface due to their amphiphilic nature. The accompanying reduction of interfacial tension ceases in a narrow concentration range (the critical micellization concentration, CMC). At concentrations above this range added surfactant will aggregate to micelles.

The first sample of the alcohol and of the hydrocarbon are dissolved in the aqueous solution not because they are soluble in water—(try to dissolve the corresponding amounts in pure water) but because they are dissolved (solubilized) into the micelles (Figs. 3B,C).

The second addition displays the difference in behavior caused by the presence of the OH group on the alcohol. The alcohol molecules will be solubilized in the interior of the micelles; and also, due to their hydrogen-bonding capacity, they may orient at the polar interface of the micelle. The hydrocarbon molecules will be solubilized in the interior of the micelle by the hydrocarbon chains of the surfactant. Since the aliphatic hydrocarbon is not capable of response to hydrogen or polar bonds from the water or the sulfate group, it will not be found close to the polar layer of the micelle. Therefore, the micelle can accept fewer hydrocarbon molecules than alcohol molecules. The excess hydrocarbon is not dissolved at all but forms macrodroplets in an emulsion which are clearly observable in the microscope.

When the alcohol is involved, the conditions will be entirely different when the maximum solubilization in the micelles is exceeded. No separation will take place to form the large droplets of an emulsion as in the case of the hydrocarbon. Instead, the interaction between the OH groups of the alcohols, the polar part of the surfactants and the water molecules is sufficiently strong to retain the three components in one phase. This phase is not a liquid, but a lamellar liquid crystal (Fig. 3D).

Such a structure is entirely different from the micellar solutions. In these, the association structures, the micelles, are small particles floating freely around in the liquid. In the liquid crystal, the association structures, the layers, fill the entire space. The optical properties will be different perpendicular to and along the layers; the liquid crystalline structure is optically anisotropic which is observed from its radiance when viewed between crossed polarizers.

Further addition of alcohol changes the liquid crystal to an isotropic liquid. From its low conductance it is obvious that the alcohol is now the continuous phase. The water is dispersed in the central part of *inverse micelles* (Fig. 3E) surrounded by the surfactant and alcohol molecules with the polar parts toward the center; the hydrocarbon parts point toward the alcohol continuous phase.

The hydrocarbon may be dissolved in this phase; the alcohol which (Fig. 3F) is the continuous liquid will dissolve hydrocarbon. The resulting alcohol/hydrocarbon solution with water and surfactant dispersed in inverse micelles is a colloidal solution; however, it is often called a *microemulsion* like an *emulsion*.

The concepts of microemulsion and emulsion are well illustrated by the contrast between the appearance of the two samples formed (1) by excess hydrocarbon in the aqueous solution of surfactant (Series II) and (2) by addition of the same amount of hydrocarbon to Series I after the final addition of alcohol. The microemulsion is transparent

since the droplet size is less than $0.1\ \mu\text{m}$, and it forms spontaneously when the components are brought together. The emulsions exemplified by some samples in Series II, Numbers 2-6 are turbid because of the large droplet size ($>1\ \mu\text{m}$). These emulsions do not form spontaneously; the sample must be shaken for the emulsion to form. If left to stand, the emulsion will separate; microemulsions of the kind described here will not.

Acknowledgments

Acknowledgment is made to the Donors of The Petroleum Research Fund, administered by the American Chemical Society, for support of the research, Grant Number ACS PRF 9934 AC5, Code Number 2149-2232.

Literature Cited

- (1) Friberg, S., *Chemtech* 6, 124 (1976).
- (2) Adamson, A. W., *J. Colloid Interface Sci.* 29, 261 (1969).
- (3) Ruckenstein, E., and Chi, J. C., *J. Chem. Soc. Faraday Trans. II* 71, 1690 (1975).
- (4) Levine, S., and Robinson, K., *J. Phys. Chem.* 76, 876 (1972).
- (5) Gerbacia, W., and Rosano, H. L., *J. Colloid Interface Sci.* 44, 242 (1973).
- (6) Shinoda, K., and Friberg, S., *Adv. Colloid Interface Sci.* 4, 281 (1975).
- (7) Friberg, S., and Buraszczenka, I., *Prog. Colloid Polymer Sci.* 63, 1 (1978).
- (8) Healy, R. N., and Reed, R. L., *Soc. Petr. Eng. J.* 14, 491 (1974).
- (9) Gillberg, G., and Friberg, S., "Evaporation-Combustion of Fuels, Advances in Chemistry Series, No. 166, (Editor: Zung, Joseph T.) Chapter 15, 221 (1978).
- (10) Advances in Liquid Crystals, Volumes I, II, and III, (Editor: Brown, Glenn H.) Academic Press, New York (1975-1978).
- (11) Biological Membranes, (Editor: Chapman, D.) Academic Press, New York (1968).
- (12) Ambrose, E. J., "Lyotropic Liquid Crystals, Advances in Chemistry Series," No. 152, (Editor: Friberg, Stig Chapter 10, 142 (1976).