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Stability Of Hydrophobic Foams

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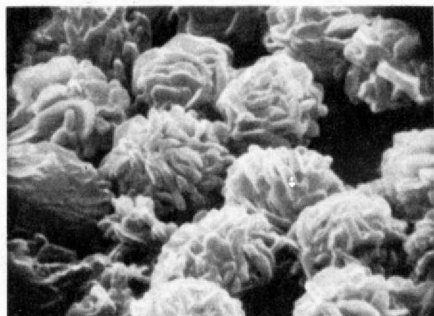
STEARIC ACID PARTICLES

Figure 4. Scanning Electron photomicrograph of stearic acid particles. The particles are formed by ultrasonic dispersions of stearic acid emulsions that are initially above the melting point of the acid (>70 °C). Note the particles are collections of plates. Magnification 6000×—average size between 4–6 μm.

come rough when the particles solidify. Stearic and palmitic acids have the roughest surfaces as can be seen by the typical example shown in Figure 4. The particles consist of a collection of crystalline plates that form on cooling from the emulsified liquid state. It is proposed that when the dimensions of the aspirities exceed a critical value that is of the same order of magnitude as the film thickness they can puncture the film and thus function as anchoring points. A similar mechanism was proposed by Van Boekel and Walstra[14] to explain how solids in the dispersed phase decrease emulsion stability. Basically, when the Laplace pressure at the tip of the curved asperity exceeds the

maximum disjoining pressure that is characteristic for the system, the film can rupture which results in particle attachment. The effect of roughness on film stability will be considered in a separate paper.

Once solid particles bridge the foam film they do not necessarily cause it to rupture. This depends on the degree of deformation and is coupled to the concentration of the surfactant. Thus, Garrett's conclusion that dynamic effects are critical is consistent with our experimental results. However, particles such as docosane and paraffin wax are almost spherical with aspect ratio close to 1. The contact angles of LAS solutions are expected to be close to those given in Table VII for hydrophobic quartz. On the basis of the requirements in Table VIII, neither docosane nor paraffin wax particles should be capable of inducing film rupture by dewetting even under dynamic conditions based on the Garrett model. Clearly, this is not consistent with the experimental results. Qualitatively, particles bridging the film appear to act as defects in the film that cause it to rupture once the film is subjected to a critical rate of deformation. We do not have a model to propose for how deformation induces rupture of such composite films and the process may involve inertial effects. Obviously, more theoretical work is required to sort out this complicated problem.

Registry No. $C_{12}EO_8$, 3055-98-9; LAS, 26248-24-8; Tergitol, 68894-53-1; quartz, 14808-60-7; palmitic acid, 57-10-3; oleic acid, 112-80-1; octadecane, 593-45-3; tetradecane, 629-59-4; docosane, 629-97-0; stearic acid, 57-11-4.

Stability of Hydrophobic Foams[§]

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The stability of foams was determined in two-phase regions containing an isotropic hydrocarbon solution and a lamellar liquid crystal. The lamellar liquid crystal showed surface activity with regard to the hydrocarbon solution. This was interpreted as due to the higher frequency of methyl groups at the surface of the liquid-crystalline state compared to the liquid state. The weaker intermolecular forces from the methyl groups were assumed to result in a lower surface tension of the liquid crystal in comparison to the liquid. This hypothesis was tested by the use of a low surface tension hydrocarbon, isooctane. No foam stability was formed in this medium, supporting the claim of the methyl groups as the surface-active element.

Introduction

The knowledge in the area of the stability of liquid films and foams has increased rapidly over the last decades as evidenced by review articles in the area.[1-8] From this basis

it appears obvious that the knowledge about aqueous foams at present is rather well developed as exemplified

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[§] Presented at the symposium on "Fluid–Fluid Interfaces: Foams", 190th National Meeting of the American Chemical Society, Chicago, IL, Sept 8–13, 1985.

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Foaming apparatus

1 Nitrogen gas
2 Regulator
3 Absorbtion bottle
4 Flow meter
5 Copper tube coil
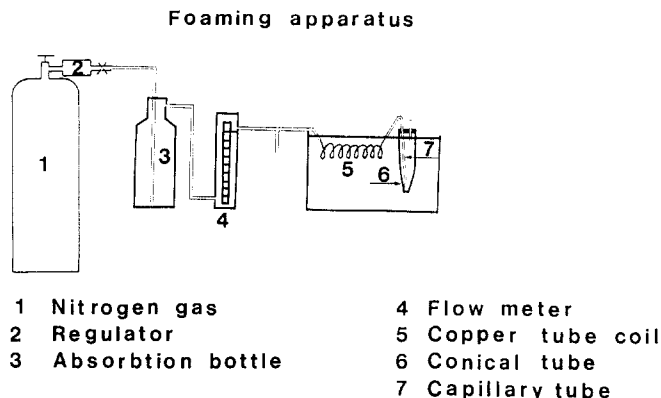6 Conical tube
7 Capillary tube

Figure 1. Experimental equipment for foaming: (1) gas cylinder; (2) pressure valve; (3) bottle to add vapor of solvents; (4) flow meter; (5) thermostated copper coil to obtain correct temperature of the gas; (6) vessel for foaming; (7) capillary.

by the recent paper by Scheludko[9] showing excellent agreement between calculated and experimental values for the critical thickness of their films.

With this fact in mind, the interest in foam stability has begun to focus on foams from more complex systems, with a less obvious basis for their stability. Ross and collaborators[10,11] have related the transient stability of nonaqueous foams from "organic" solvents to the upper consolute point in solvent mixtures. Neustadter[12] has evaluated the complex background of the stability of foams from crude oils and Friberg,[13,14] Wasan,[15] and Petrov[16] have investigated the influence of long-range order structures on foam stability. These latter results with solubilized water laid the foundation for the formulation of a completely nonaqueous vehicle to produce a foam with excellent stability.[17] This nonaqueous foam consisted of a hydrocarbon solution and a lamellar liquid crystal and the reason for its stability should be identical with the one for the corresponding systems in hydrophobic foams.[13-16]

We have earlier[17] suggested that the surface activity of the liquid-crystalline phase vs. a hydrocarbon solution may be due to the concentration of methyl groups at its surface. A surface of CH_3 groups has lower surface tension ($\simeq 23$ mN/m) than that of a liquid hydrocarbon in which the methylene groups are frequently exposed at the surface raising the surface tension by their high interaction.[18] A simple test of this hypothesis would be to compare the foam stability in systems with benzene ($\gamma \simeq 30$ mN/m) with that in isooctane systems ($\gamma \simeq 20$ mN/m).[19] Induced foam stability by the presence of a liquid crystal in the former systems and none in the latter ones would corroborate the assumption.

Figure 2. Part of the phase regions in the system water/tetraethylene glycol dodecyl ether (TEGDE)/benzene. Compositions of foamed samples are marked 1-4. The line through the TEGDE/benzene solubility area shows compositions which were used for surface tension measurements. L_2 = inverse micellar solution; D = lamellar liquid crystal.

This article presents partial phase diagrams with water, hydrocarbon, and a nonionic surfactant and variation of foam stability in the two-phase regions hydrocarbon/surfactant isotropic solution–lamellar liquid-crystalline phase.

Experimental Section

Materials. The surfactant was tetraethylene glycol mono-*n*-dodecyl ether (TEDGE), 99.5% according to gas chromatography test from Nikkol. The hydrocarbons were 2,2,4-trimethylpentane (isooctane), 99%, benzene, 99%, and hexadecane, 99% (all three from Aldrich). The water was twice distilled. All chemicals were used as supplied.

Determination of the Solubility Region. The region for inverse micellar solution in the surfactant/hydrocarbon/water system at 25 °C was determined by titration with water and /or hydrocarbon. The border lines were checked by long-time storage of samples. The liquid-crystalline phase was identified by optical microscopy using polarized light. The change from isotropic solution to liquid crystal was followed by analysis of two-phase samples separated by centrifugation.

The apparatus for determining foam stability is shown in Figure 1. Nitrogen gas was passed through a regulator into an absorption bottle containing the volatile component of the foam to ensure vapor saturation before foaming. The solvents were hydrocarbon and water.

The foaming gas was passed through a flow meter, a copper tube coil to achieve correct temperature, 25 °C, and ultimately a capillary tube into the solution being foamed. The diameter of the capillary tube was changed to give equally sized bubbles for the different samples. A conical tube with a total volume of 50 mL was chosen as the foam container. One milliliter of each solution was foamed to a volume of 50 mL or until liquid was no longer available.

The foam volume was measured as a function of time after stopping the air flow.

The samples for foaming were prepared from separated phases within the two-phase region.

Measurement of Surface Tension. Surface tension measurements were made by using a du Noüy balance with a platinum ring. The ring was cleaned between determinations by heating in a gas burner flame followed by a rinse with distilled water and with absolute alcohol.

Results

Phase regions for the part of the diagram used in the investigations are given in Figures 2–4. In Figure 2, we find a liquid isotropic solution of tetraethylene glycol

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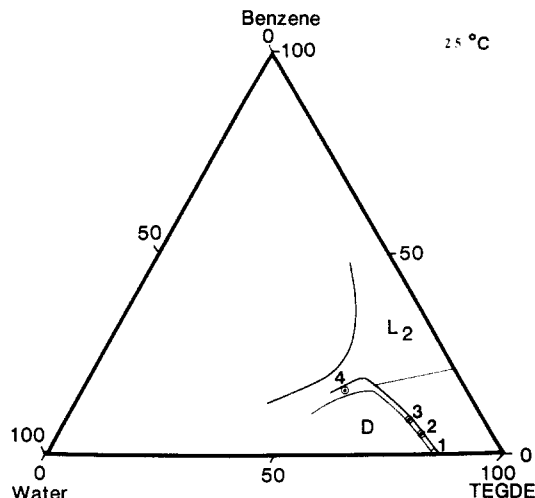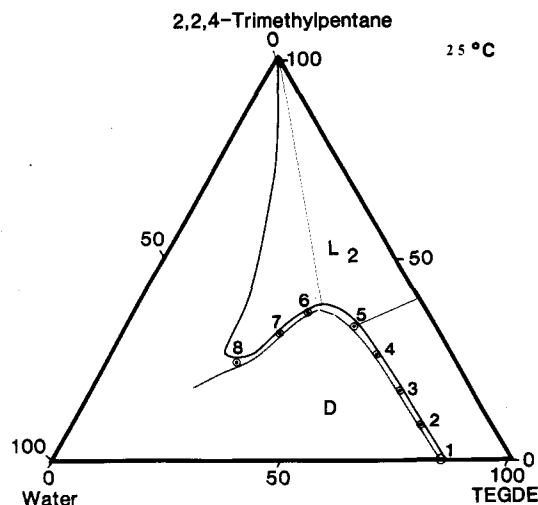(19) Kunieda, H.; Hanrin, M. *J. Chem. Soc. Jpn.* **1979**, *5*, 561.

Figure 3. Part of the phase regions in the system water/2,2,4-trimethylpentane/tetraethylene glycol dodecyl ether (TEGDE). Compositions of foamed samples are marked 1–8. The line through 2,2,4-trimethylpentane solubility area shows compositions which were used for surface tension measurements. L_2 = inverse micellar solution; D = lamellar liquid crystal.
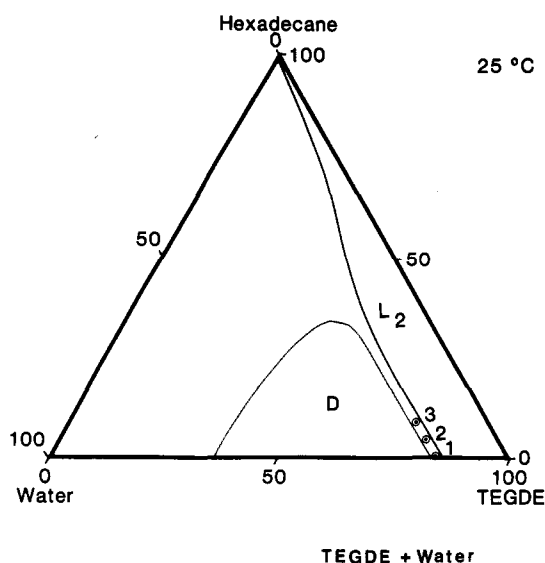

Figure 4. Part of the phase regions in the system water/tetraethylene glycol dodecyl ether/hexadecane compositions of foamed samples are marked 1–3. L_2 = inverse micellar solution; D = lamellar liquid crystal.

dodecyl ether and benzene with solubilized water to the right in the diagram. At an approximate hydrocarbon ratio of 2.3:1 a strong solubilization of water was found giving a narrow channel of isotropic solution pointing toward the aqueous corner. The liquid-crystalline phase was found close to the left lower part of the isotropic liquid-phase region border toward the water. The samples used for foaming are marked in the diagram as are the compositions used for surface tension measurements. The corresponding diagram for the system with 2,2,4-trimethylpentane as hydrocarbon is foun in Figure 3. In this case, the isotropic liquid solution area shows an increased water solubilization for a higher hydrocarbon/surfactant ratio. This means that the liquid-crystalline phase now reaches to higher hydrocarbon/surfactant ratios than for the system with benzene. The compositions used for foaming experiments are marked in the two-phase area of the isotropic liquid solution plus liquid-crystalline phase. The compositions used for determination of surface tensions are also marked in the diagram.
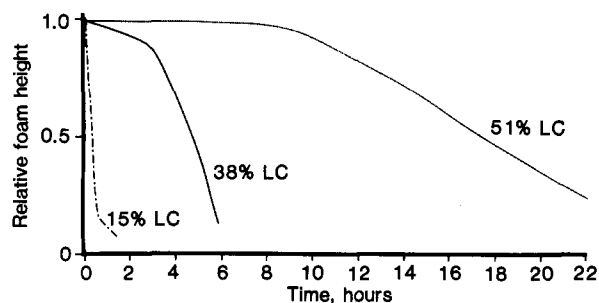

Figure 5. Relative foam height as a function of time for tetraethylene glycol dodecyl ether (TEGDE) and water at 25 °C (sample 1, Figure 2). The number given shows percentage of liquid-crystalline phase in the foamed mixture.
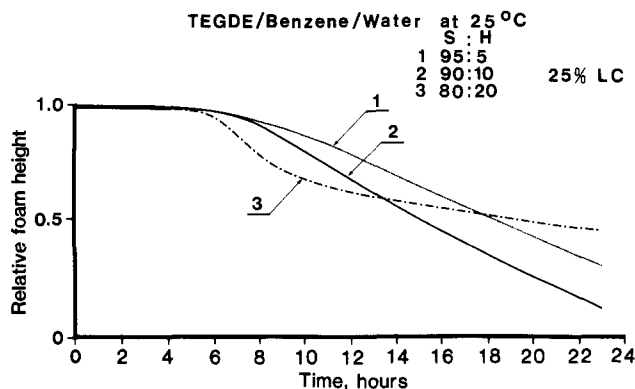

Figure 6. Relative foam height as a function of time for tetraethylene glycol dodecyl ether (TEGDE)/benzene/water system at 25 °C with 25% liquid crystalline and varied benzene ratios. (1) TEGDE:benzene = 95:5 (sample 2, Figure 2); (2) TEGDE:benzene = 90:10 (sample 3, Figure 2); (3) TEGDE:benzene = 80:20 (sample 4, Figure 2).

Figure 4 shows part of the phase diagram with hexadecane as the hydrocarbon. In this case no increase in the solubilization of water was found.

The first two diagrams are characteristic of combinations at temperatures in excess of the phase-inversion temperature (PIT),[20] while the third one is below this value.

Foam Stability. The foam stability of tetraethylene glycol/water combinations selected within the two-phase range of the lamellar liquid crystal (D) and the isotropic solution (L_2) on the water surfactant axis are shown in Figure 5. The results clearly demonstrate influence of the amount of liquid-crystalline phase being present on foam stability. With 15% liquid-crystalline phase, the foam height was reduced to half its value within a time frame of 15 min; for 38% liquid-crystalline phase we find the corresponding time about 5 h. For the system with 51% liquid-crystalline phase, the corresponding time was approximately 18 h. It should be observed that there is also a difference in the destabilization pattern for the three compositions. In the first sample, with a small amount of liquid-crystalline phase, the destabilization starts immediately and proceeds approximately as an exponential function of time. With the intermediate concentration of liquid-crystalline phase, we find an initial period of slow breakdown followed by a faster period after about 3 h. For the highest amount of liquid-crystalline phase, 51%, the slow breakdown now lasts for 10 h. With these results as a basis, the foams were investigated in the two-phase area in the benzene system (Figure 2). Several combinations with varying liquid-crystalline phase amounts were used in order to establish a pattern. From these results, the
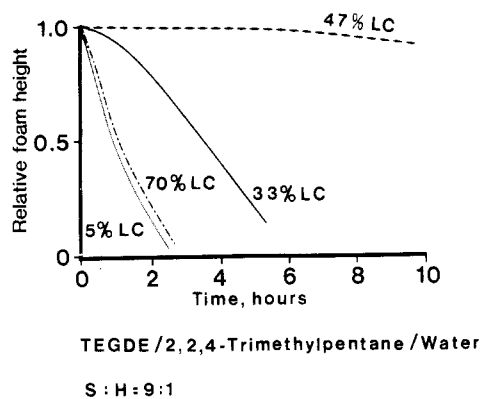
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TEGDE/2,2,4-Trimethylpentane/Water

S : H = 9 :1

Figure 7. Relative foam height as a function of time for tetraethylene glycol dodecyl ether (TEGDE)/2,2,4-trimethylpentane/water at 25 °C. Surfactant:hydrocarbon = 9:1 (sample 2, Figure 3). The numbers are liquid-crystalline content of the foamed mixture.
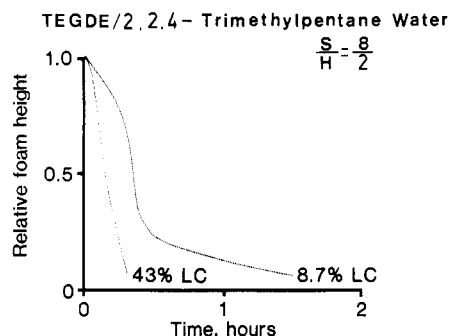

Figure 8. Relative foam height as a function of time for tetraethylene glycol dodecyl ether (TEGDE)/2,2,4-trimethylpentane/water at 25 °C. Surfactant:hydrocarbon = 8:2 (sample 3, Figure 3).

foam stability for 25% liquid crystalline phase has been selected in order to illustrate the dependence of the hydrocarbon content. Foam stability for surfactant hydrocarbon ratios of 17, 9, and 4.0 are shown in Figure 6, marked 1, 2, and 3; sample compositions are marked 2, 3, and 4 in Figure 2. The ratio of 4.0 was chosen in order to be able to study foam stability in the part of the two-phase area where high water solubilization is found. The results, Figure 6, clearly show the addition of benzene to cause an enhancement of foam stability compared to the sample without benzene present, Figure 5.

The results for the isooctane system showed considerably greater variation with the amount of hydrocarbon present, and these results are presented in more detail. For the surfactant/isooctane ratio of 9:1 (sample 2 in Figure 3), the dependence of the amount of liquid-crystalline phase is given in Figure 7. The stability of the foams with this low content of the hydrocarbon was comparable to the one obtained for systems without hydrocarbons present, Figure 5. The surfactant/hydrocarbon ratio of 4 (sample 3, Figure 3), on the other hand, gave considerably less foam stability as shown by Figure 8. The half-life of the foam is now reduced to a matter of less than 1 h and the amount of liquid-crystalline phase has little influence. The stability of a foam with 8.7% liquid-crystalline phase and 43% liquid-crystalline phase is approximately the same. For higher hydrocarbon surfactant ratios (sample 4, Figure 3), the foam stability was even more reduced, Figure 9. The total breakdown on the foam was now a matter of minutes. The total breakdown time for the surfactant/hydrocarbon ratio 1.6 (sample 5, Figure 3) took place in less than 1 min, Figure 10. For compositions in the higher water solubilization area marked 6, 7, and 8 in Figure 3, there was no
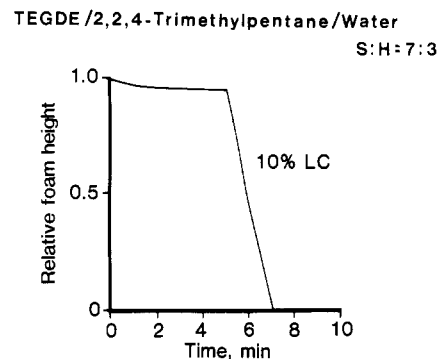
TEGDE/2,2,4-Trimethylpentane/Water

S:H=7:3

Figure 9. Relative foam height as a function of time for tetraethylene glycol dodecyl ether (TEGDE)/2,2,4-trimethylpentane = 7:3 (sample 4, Figure 3) at 25 °C with 10% liquid crystal.

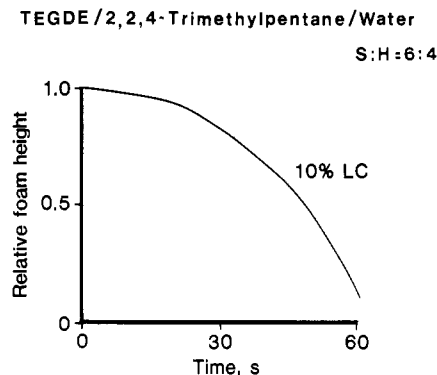TEGDE/2,2,4-Trimethylpentane/Water

S:H=6:4

Figure 10. Relative foam height as a function of time for tetraethylene glycol dodecyl ether (TEGDE)/2,2,4-trimethylpentane = 6:4 (Sample 5, Figure 3) at 25 °C with 10% liquid crystal.
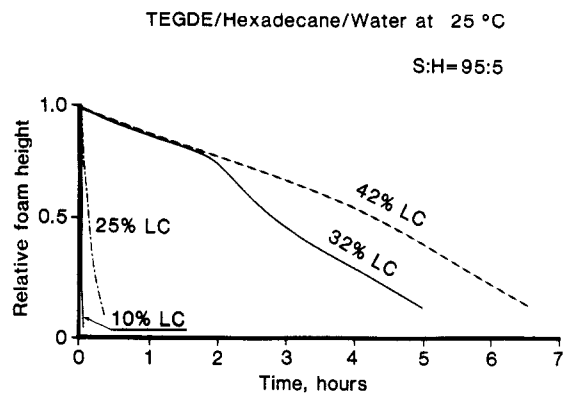
TEGDE/Hexadecane/Water at 25 °C

S:H=95:5

Figure 11. Relative foam height as a function of time for tetraethylene glycol dodecyl ether (TEGDE)/hexadecane/water at 25 °C. TEGDE/hexadecane = 95:5 (sample 2, Figure 4). The numbers are liquid crystalline content of the foamed mixture.
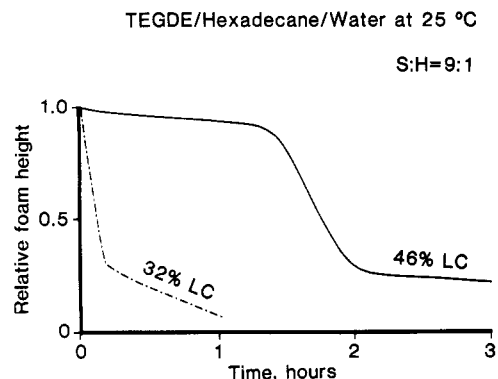
TEGDE/Hexadecane/Water at 25 °C

S:H=9:1

Figure 12. Relative foam height as a functin of time for tetraethylene glycol dodecyl ether (TEGDE)/hexadecane/water at 25 °C. TEGDE/hexadecane = 90:10 (sample 3, Figure 4). The numbers are liquid crystalline content of the foamed mixture.
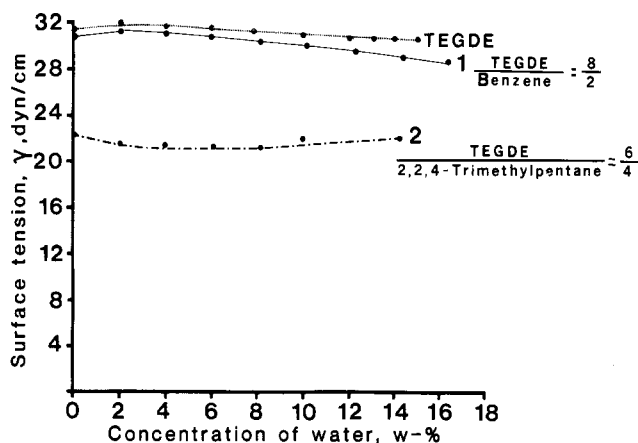
Figure 13. Surface tension of pure tetraethylene glycol dodecyl ether (TEGDE) and TEGDE with hydrocarbon as a function of water concentration.
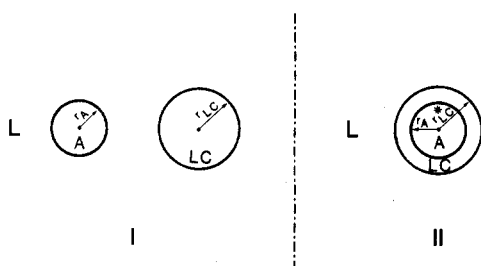

Figure 14. Foam bubble and the liquid crystal may be separated (I) or the liquid crystal may cover the air bubble (II).

observable foam stability at all. Figures 11 and 12 show the foam stability with hexadecane as the hydrocarbon being of the same magnitude as the one with the 2,2,4-trimethylpentane.

The surface tension for compositions along the lines marked in Figures 2 and 3 are given in Figure 13 .

Discussion

The results confirm earlier suggestions[13-17] of the presence of a liquid-crystalline phase as essential for the stability of a foam in a hydrocarbon. In addition, the investigations also provide information about the mechanism of the stabilization.

The liquid-crystalline phase is preferentially located between the gas and the liquid isotropic hydrocarbon solution.[17] This fact indicates the liquid crystal to possess a lower surface tension than the corresponding solution of similar composition. The calculations by Gruen[21,22] have shown the methyl groups to be preferentially located at the surface of the liquid crystal. By combination of this information with the results by Zisman,[18] showing the methyl groups to have surface tension of approximately 23 mN/m while the methylene groups show a value of 28 mN/m, an assumption that the lower surface tension of the liquid crystal is due to the higher frequency of methyl groups at its surface in comparison with the isotropic solution can be made.

This condition can be readily derived in the following way. We wish to compare the configuration in Figure 14, part I, with an air bubble and a liquid crystalline droplet suspended in the isotropic solution, with the same volumes forming the configuration in Figure 14, part II, where the
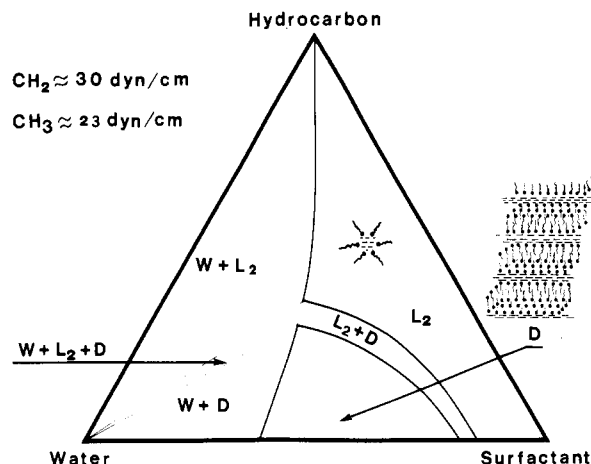
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Figure 15. Increased water content changes the association structure from inverse micelle L_2 to lamellar liquid crystal (D).

liquid crystal is spread at the interface. Realizing that for a nonequilibrium system like this, the dominant driving force for the formation of II from I is the difference in total surface tension, one obtains the condition

$$r_A^2\gamma_{L/A} + r_{LC}^2\gamma_{L/LC} > r_A^2\gamma_{A/LC} + r^*_{LC}^2\gamma_{LC/L} \quad (1)$$

Since the volumes in I and II are equal, $r^*_{LC} = (r_{LC}^3 + r_A^3)^{1/2}$ and hence

$$r_A^2\gamma_{A/LC} < r_A^2\gamma_{L/LC} - \gamma_{L/LC}[(r_{LC}^3 + r_A^3)^{2/3} - r_{LC}^2] \quad (2)$$

Writing $r_{LC} = kr_{A'}$ gives

$$\gamma_{A/LC} < \gamma_{L/A} - \gamma_{L/LC}[(k^3 + 1)^{2/3} - k^2] \quad (3)$$

The factor within square brackets is ~ 1 for small k, that is, when the air bubble is large compared to the thickness of the layer of liquid crystal. (3) then reduces to the trivial case of simple spreading pressure for which

$$\gamma_{LC/A} < \gamma_{L/A} - \gamma_{L/LC} \quad (4)$$

is the condition for infinite spreading of the LC on a planar L surface. When k is large, the thickness of the liquid-crystalline layer is comparable to the radius of the bubble. The perturbation of the liquid-crystalline structure by the air bubble is small and the last term in (3) tends to zero, i.e., the condition becomes

$$\gamma_{LC/A} < \gamma_{L/A} \quad (5)$$

Irrespective of the geometric conditions, we have

$$\gamma_{L/LC} < \gamma_{L/A} \quad (6)$$

and, hence, the essential condition for spontaneous location at the interface is given by eq 5; i.e., the liquid-crystalline phase should have a lower surface tension than the liquid toward air.

In the present case this difference in surface tensions is caused by the addition of water. However, as shown by Figure 13, addition of water does not increase the surface tension of the L_2 solution. The effect of the increased volume of water is a change in the association structure from reversed micelles to a lamellar liquid crystal (schematically shown in Figure 15).

The reverse micelles are dynamic structures which do not preferentially expose the methyl groups in such a way that a low-energy surface is formed, as is clearly verified by the absence of a reduction in the surface tension in TEGDE or TEGDE/benzene solutions as water is added (Figure 13). However, when the addition of water is extended so that lamellar phase is formed, an ordered

structure with preferential exposure of methyl groups is introduced. Instead of a situation in which there is no vehicle present with sufficiently low surface energy to be surface active, now the methyl groups with a surface tension of 23 dyn/cm (18) are concentrated to the surface. Methylene groups would give a higher surface tension, 28–30 dyn/cm.[18]

This means that the surface activity of the lamellar liquid crystal which is induced by the long-range ordering will not function against a hydrocarbon with sufficiently low inherent surface tension. The gradually reduced foam stability with increased aliphatic hydrocarbon content illustrates this fact. As shown by Figure 13, addition of water to such hydrocarbons does not increase the surface tension. As 2,2-4-trimethylpentane is added to TEDg-DE/water, the surface tension is gradually reduced and when it reaches a critical point, no foam stability is left. A similar mechanism acts for hexadecane but in this case the low vapor pressure may also lead to some liquid hydrocarbon being present at the surface to air.

In the latter case with a hydrocarbon with a low vapor pressure, the possibility of a layer of solubilized hydrocarbon remaining on the surface of the liquid crystal should also be taken into account. The nature and structure of that layer is unknown.

The results strongly indicate the presence of the liquid-crystalline phase at the surface as the decisive element in the stabilization of the foam but leave no information about the stabilizing action of the liquid-crystalline layer.

As a first item, it is essential to point out that the Ross[11,12] results show an opposite result for a phase separation with two liquids. The surface free energies are of similar magnitude for the liquid crystal and the liquid, and, hence, it appears reasonable to assume that the enhanced rigidity of the liquid-crystalline phase is responsible for the improved stability of the present foams. In accordance with earlier literature[23] a monomolecular layer with high vicosity is a sine qua non for the stability of aqueous foams. A surface multimolecular layer of high viscosity should presumably fill the same function.

In addition, the prevention of drainage by the viscous liquid-crystalline phase is also an important factor. Our earlier contributions[4] demonstrated the enrichment of liquid-crystalline phase in the foam and slower drainage. However, the final conclusions must await further results.

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Method for Assessment of Fetal Lung Maturity[⊥]

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A physicochemical method for evaluation of fetal lung maturity is described. The method is based on the possibility for spontaneous formation of a stable, with respect to rupture, black foam film (BFF) from mature amniotic fluid (AF). The stable BFF from AF is obtained as a result of a spontaneous processes taking place at the air/liquid interface, the interface which lines the alveolus. While mature AF samples give BFF that are stable for a long time, the foam films from immature AF samples are not stable and invariably rupture under the same conditions. Thus, failure to obtain a stable BFF from AF samples indicates a high risk for development of respiratory distress syndrome (RDS) in the newborn. The technique for detecting foam film rupture and BFF formation from AF samples is microscopic. The results obtained by the new method in a study of 182 samples from normal pregnancies are compared with those from the clinical investigation as well as with the lecithin/sphingomyelin (L/S) ratio of the samples. The BFF method proves to be of a higher diagnostic potential than the L/S ratio; moreover it is fast, is easy to perform, produces simple and unambiguous results, and requires minimal sample volumes.

Introduction

A series of papers have shown the correlation between lung immaturity and respiratory distress syndrome (RDS).[1,2] A number of methods for prediction of fetal lung maturity by examination of samples from amniotic fluid have been developed (e.g., ref 3–11). Still, "no single test of amniotic fluid has yet been found to be completely reliable, easily performed, and universally applicable".[12]

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