Optimum Formulation of Surfactant/Water/Oil Systems for Minimum Interfacial Tension or Phase Behavior

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ABSTRACT

A screening test used to help select surfactant systems potentially effective for oil recovery is to identify those formulations that yield middle-phase microemulsions when mixed with sufficient quantities of oil and brine. A correlation is presented to link these variables regarding their contributions to middle-phase formation: structure of the sulfonated surfactant, alkane carbon number (ACN), and alcohol type and concentration. WOR and temperature effects are introduced as correction terms added to the empirical correlation.

Sets of variables that give middle-phase microemulsions are shown as identical to those defining the low tension state without observable middle phases. This generally occurs for low surfactant concentrations.

INTRODUCTION

Healy and Reed[1] and Healy *et al.*[2] have shown that the phase behavior of surfactant/brine/oil systems is a key factor in interpreting the performance of oil recovery by microemulsion processes. By systematically varying salinity, they found low interfacial tensions and high solubilization of both oil and water in the microemulsion phase to occur in or near the salinity ranges giving three phases. Since both low interfacial tensions and a high degree of solubilization are considered desirable for oil recovery, the conditions for three-phase formation assume added importance. Similar conclusions have been reported in other recent papers.[3-5]

Several investigators have considered the effect of different variables on the range of salinities for which three phases form. This optimum salinity (a more precise definition is given in a subsequent section) has been found to decrease with increasing surfactant molecular weight,[6] and to increase with increasing chain length of the alcohol cosurfactant.[7] Studies on the effect of alcohols by Jones and Dreher[8] and Salter[9] provided results similar to those reported by Hsieh and Shah.[7]

The interfacial tension at surfactant concentrations low enough so that a discernible third phase does not form has been the subject of considerable investigation regarding surfactant molecular weight and structure, oil ACN, salinity and surfactant concentration, and alcohol addition.[14-16] A recent paper[10] was a first attempt to tie together the low tension state observed at low surfactant concentrations and the three-phase region observed at higher surfactant concentrations. All indications point to an inextricable intertwining of phase behavior, surfactant partitioning, solubilization, and low tensions. This paper corroborates the equivalence of three-phase behavior and minimum tension as criteria for optimum formulation and presents a correlation that quantifies the trends observed previously.

EXPERIMENTAL

Aqueous phases containing surfactant, electrolyte (NaCl), and alcohol were contacted with an oil phase by shaking and allowed to stand until phase volumes became time independent for 2 days. All concentrations are expressed in grams of chemical per deciliter of aqueous phase (gpdl) before contacting with the hydrocarbon phase. Unless otherwise noted, the oil phase represents 20% of the initial total volume. All measurements, unless otherwise noted, were conducted at room temperature (25 ± 1ºC).

Data are reported for a variety of surfactants: Exxon C_9, C_{12}, and C_{15}™ orthoxylene sulfonates in the sodium and monoethanol amine salts; Witco TRS 10-80™; Monsanto Chemical Co. alkylbenzene sulfonates; Alcolac Siponate DS-1O™; an isomeric mixture of sodium dodecylbenzene sulfonate species; 5 (para ethylphenol) decyl sulfonate synthesized in our laboratory using Doe *et al.'s* procedure.[11]

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CHARACTERIZATION OF OPTIMAL FORMULATIONS

The phase behavior of surfactant/oil/brine systems may be represented advantageously on a ternary diagram, assuming that the salt and water partition between phases in equal proportions.[1,2] Most investigators have elected to use a pseudoternary diagram in which alcohol is presumed to partition in a predetermined way. Some authors[3,7] have represented the surfactant and cosurfactant at one vertex, which is valid if the surfactant and cosurfactant partition in the same ratio (for possible difficulties, see Hsieh and Shah[7] and Salter[9]). Since the phase behavior can be extremely sensitive to alcohol concentration and not to surfactant concentration, the representation of both values at a common vertex can magnify minor partitioning differences.

A more restricted approach is to consider the cosurfactant as an independent formulation variable.[12] A complete respresentation would require a ternary-phase diagram for each alcohol concentration. This approach assumes that alcohol partitioning is independent of other formulation variables and therefore, is, an approximation. The approach has two advantages: (1) it allows the alcohol effect to be separated from the effect of surfactant concentration, which vanishes for isomerically pure surfactants[13]; and (2) the representation may be particularly useful for high molecular weights (low molecular weight alcohols) that tend to partition strongly into the oil phase (remain in the aqueous phase).

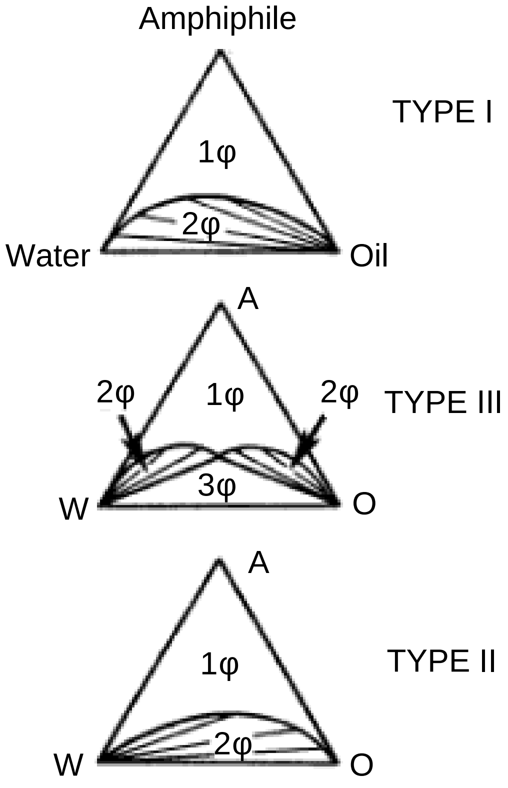

FIG. I - TERNARY DIAGRAM TYPES FOR AMPHIPHILIC COMPOUND/WATER/OIL SYSTEMS.

Fig. 1 shows the pseudoternary diagram to be considered here. The three vertexes correspond to the amphiphile (surfactant), brine, and oil, respectively. The surfactant may be a mixture of sulfonates, but cosurfactants such as alcohols are excluded. For the experiments reported here, the oil vertex represents a pure alkane characterized by its ACN. The brine vertex represents an aqueous NaCl solution characterized by its salinity, S. Three general types of systems are to be considered (Fig. 1). Type I systems are those for which the multiphase region has a lower-phase microemulsion in equilibrium with excess oil for all WOR's. Type II systems are upper-phase microemulsions in equilibrium with excess brine. Type III systems are those that exhibit a middle-phase microemulsion for some WOR's. Type III systems also exhibit two-phase regions for some WOR's (Fig. 1).

The variables to be considered here are classified in three groups: (1) *formulation* variables are those factors related to the components of the system - surfactant structure, oil carbon number, salinity, and alcohol type and concentration are all considered to be formulation variables; (2) the only *external* variable considered here is temperature - pressure has not been studied although it may have an effect under extreme conditions; and (3) two *Position* variables also are needed to locate the system on the ternary diagram - surfactant concentration and oil-water ratio will suffice here.

By systematically changing one of the nine variables while keeping the others constant (called a scan), the system may exhibit different phase behavior. Table 1 shows the qualitative trends that may be expected when making a scan.

Ideally, changing any one of the position variables - surfactant concentration or WOR - should not tend to change the system type; however, a complex effect can result (Table 1). Changing the WOR tends to change the cosurfactant concentration in both the oil- and water-rich phases, which in turn can change the type of system. Complex changes are observed when varying the surfactant concentration. In some cases, particularly at low surfactant concentrations, the intermediate Type III

TABLE 1 - QUALITATIVE EFFECT OF ALL VARIABLES ON THE OBSERVED PHASE BEHAVIOR OF ANIONIC SURFACTANTS

Scanned Variables (increase)	Ternary Diagram Transition (types)
Salinity	I → III → II
ACN	II → III → I
Temperature	II → III → I
High molecualr weight alcohol	I → III → II
Surfactant hydrocarbon chain length	I → III → II
WOR	Not Avaible
Surfactant Concentration	Complex

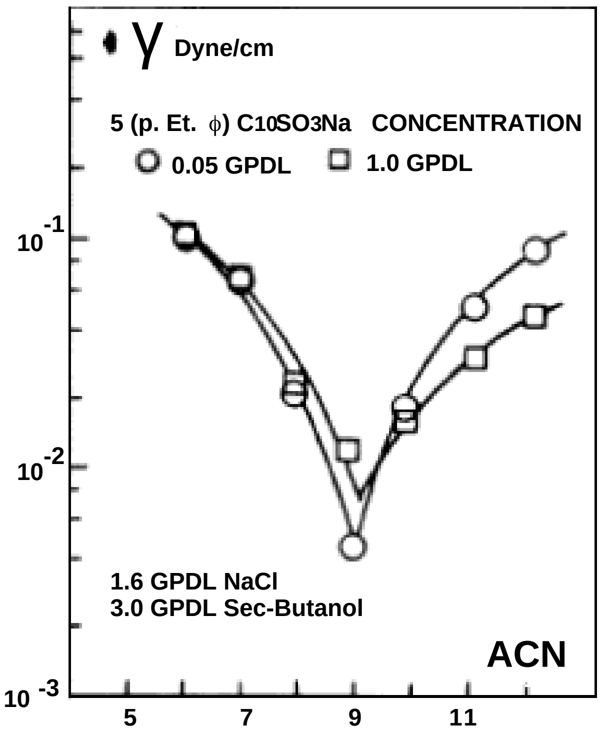

FIG.2 - INTERFACIAL TENSION VS OIL ACN FOR AN ISOMERICALLY PURE SURFACTANT (NO CONCENTRATION DEPENDENT).

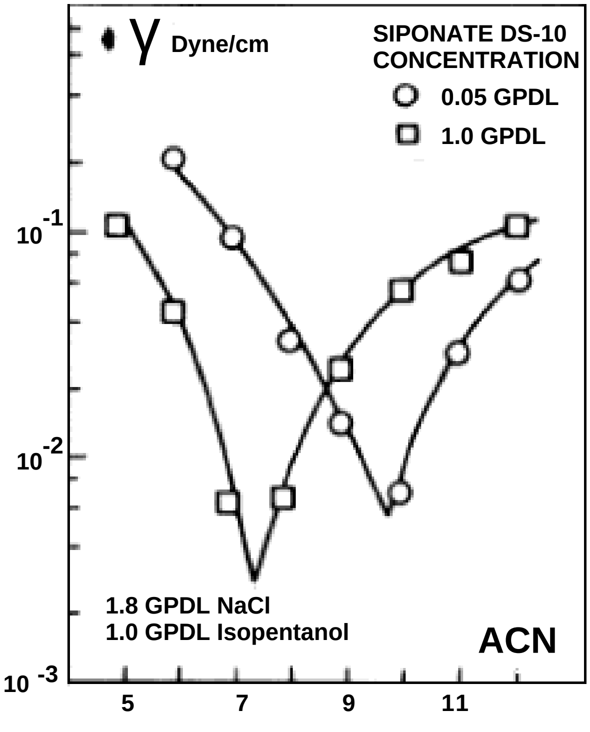

FIG. 3 - INTERFACIAL TENSION VS OIL ACN FOR COMMERCIAL DODECYL BENZENE SULFONATE (CONCENTRATION DEPENDENT).

system is not observed; instead, a direct Type I ↔ II is found that is characterized by a minimum interfacial tension. Fig. 2 shows data that reinforce the principle that the optimum formulation obtained from minimum tension at low surfactant concentration and no detectable three-phase occurrence is the same as the optimum formulation obtained at high surfactant concentration from both three-phase behavior and minimum tension. Note that this comparison must be conducted with an isomerically pure surfactant to avoid the concentration shift (the preferred hydrocarbon depends on concentration) exhibited by mixtures of surfactants.[10] As an example of this complication, Fig. 3 shows that for a commercial isomerized dodecyl benzene sulfonate, a significant shift may be caused by concentration.

OPTIMUM SALINITY AND ACN

To investigate the relationship between salinity and the molecular weight of the oil, salinity scans were conducted for a series of alkanes, while maintaining all other variables constant. Typical results are shown in Fig. 4. The shaded area corresponds to the region for which three phases are observed. The trends shown in Fig. 4 are consistent with the observations of other investigators7 and are typical of many systems studied in our laboratory.[12,13] This paper correlates this observed behavior. To simplify the task, it is convenient to define an optimum salinity, S*, associated with each alkane, rather than to try to deal with a range of salinities for which middle phases exist. The optimal salinity is taken here as the average salinity for middle-phase formation, all other variables being held constant. Other definitions are possible,[12] but these do not differ significantly from the one adopted here.

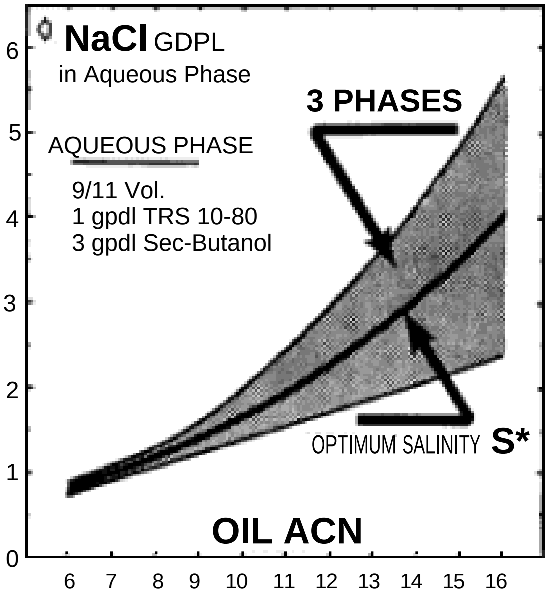

FIG. 4 - PHASE DIAGRAM SALINITY-ACN, SHOWING THE OPTIMUM SALINITY CURVE S*.

Fig. 5 shows the logarithm of the optimal salinity for different alcohol formulations as a function of ACN. Figs. 6 and 7 contain similar plots for other surfactant and alcohol formulations. Many other systems have been tested and all have yielded similar results.[12] Interestingly, all curves are straight parallel lines. We then can assert that

$$\text{Ln } S^* = K\,(\text{ACN}) + \ldots \qquad (1)$$

where $K = 0.16 \pm 0.01$ when S^* is expressed in grams of NaCl per 100 cm^3 (gpdl). K has this constant value for all alkyl aryl sulfonates tested. The dots in the equation represent terms that depend on the other variables. Earlier studies of salinity effects on the low tension regime with no middle phase[14] can be used for comparison, giving $K = 0.16 \pm 0.05$.

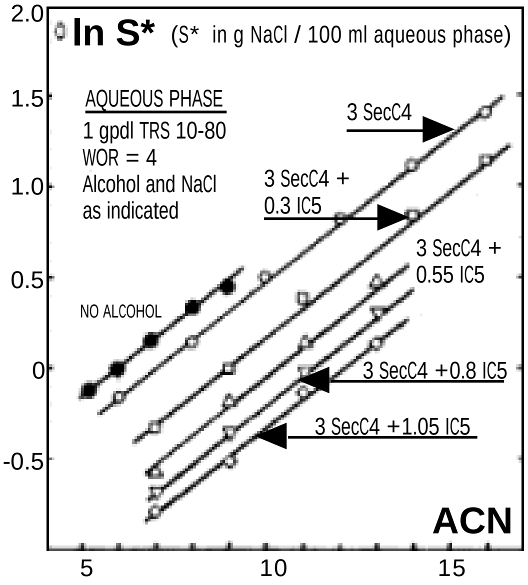

FIG. 5 - CORRELATION ln S* vs ACN AT DIFFERENT ALCOHOL CONCENTRATIONS (SEC C4 = SEC-BUTANOL; IC5 = ISOPENTANOL) FOR A COMMERCIAL PETROLEUM SULFONATE.

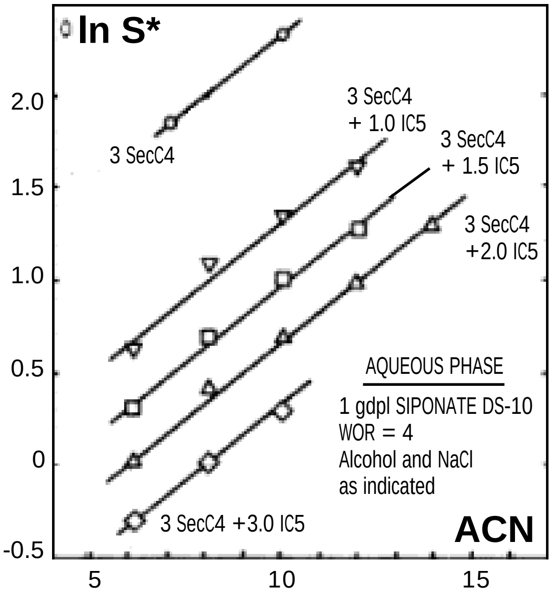

FIG. 6 - CORRELATION ln S* vs ACN AT DIFFERENT ALCOHOL CONCENTRATIONS FOR A COMMERCIAL DODECYL BENZENE SULFONATE.

ALCOHOL AND SURFACTANT TERMS

Figs. .5 through 7 show that for a given surfactant, the position of the straight lines shifts with alcohol type and concentration. With increased isopentanol concentration, the straight line shifts downward (or right) on the graph. Let the vertical shift caused by the addition of alcohol (i.e., the vertical distance of the straight line from the position for alcohol-free systems) be called f(A). Thus, by this definition, f(A) vanishes when the alcohol concentration tends to zero.

To be independent of the other variables, f(A) should be the same for different surfactants, ACN's, and salinities. Since it is not always possible to measure the interfacial tension or to form middle phases in the absence of alcohol, particularly at high salinities, a secondary standard for f(A) is - 0.16 for 3 gpdl of sec-butanol. Figs. 8 through 10 show that f(A) essentially is independent of the other variables for the alcohols tested (N- and isopentanol, N-hexanol, and secondary butanol). However, this is not the case for lighter alcohols, such as N-butanol, for which a more sophisticated approach will be needed. We will consider here only the case of high molecular weight alcohols that we believe partition preferentially into the oil phase. Then, f(A) depends only on alcohol type and concentra-

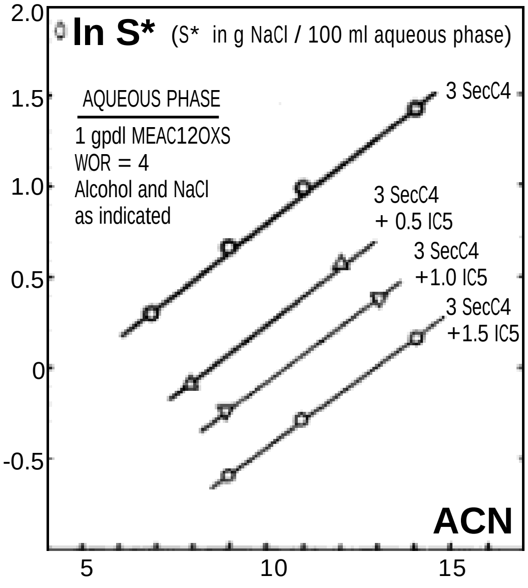

FIG. 7 - CORRELATION ln S* vs ACN AT DIFFERENT ALCOHOL CONCENTRATIONS FOR THE MONOETHANOL AMINE SALT OF DODECYL ORTHOXYLENE SULFONATE.

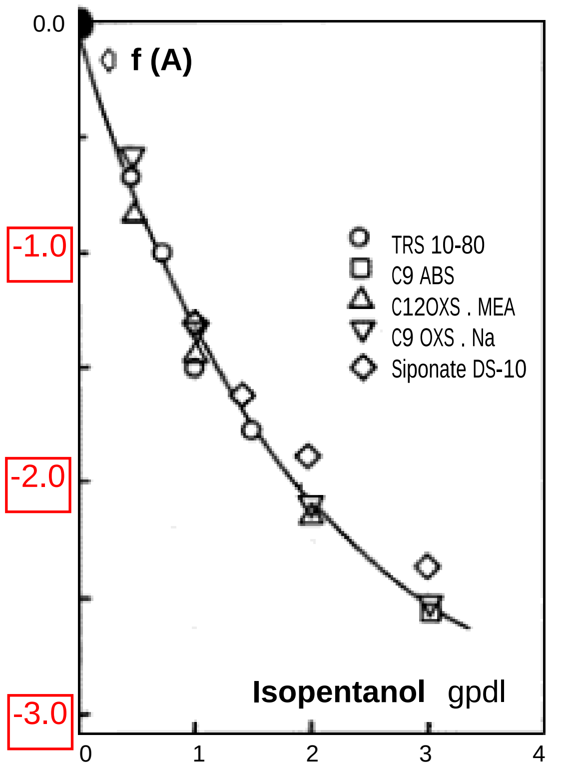

FIG. 8 - f(A) VS ISOPENTANOL CONCENTRATION FOR SEVERAL SURFACTANTS.

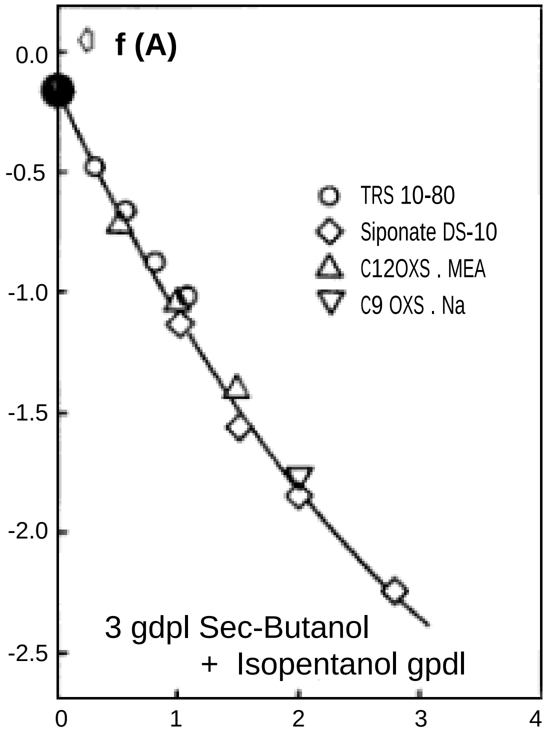

FIG. 9 - f(A) FOR 3 gdl SEC-BUTANOL PLUS VARIOUS CONCENTRATIONS OF ISOPENTANOL.

tion. In this case, the correlation may be written as

$$\ln S^* = K\,(ACN) + f(A) + \ldots \qquad (2)$$

where the dots stand for terms that depend on the surfactant structure, temperature, and position variables. Let us define a parameter characteristic of the surfactant, σ, so that

$$\ln S^* = K\,(ACN) + f(A) - \sigma \ldots \qquad (3)$$

under the experimental conditions, i. e., at 25ºC, WOR = 4 and surfactant concentration = 1.0 gpdl.

Fig. 11 shows the ln S* - ACN plot for three different surfactants at the same conditions. If S* and ACN are measured, K = 0.16, and f(A) = - 0.16 for 3 gpdl sec-butanol, then it is possible to calculate the value of the characteristic parameter σ, for each surfactant.

EPACNUS AND N_{min}

Consider the point of the correlation corresponding to no added alcohol and unit salinity; at this point, f(A) = 0 and ln S* = 0; thus, from Eq. 3,

$$ACN = \sigma/K \ldots. \qquad (4)$$

In other words, the preferred oil ACN is σ/K. This ACN value is called Extrapolated Preferred Alkane Carbon Number at Unit Salinity (EPACNUS). The EPACNUS is essentially a generalization of the N_{min} concept reported previously.[10,16] Fig. 12 shows how to obtain the EPACNUS for a given surfactant, if one optimum formulation is known. The EPACNUS may not be actually measured directly, but in any event, it can be

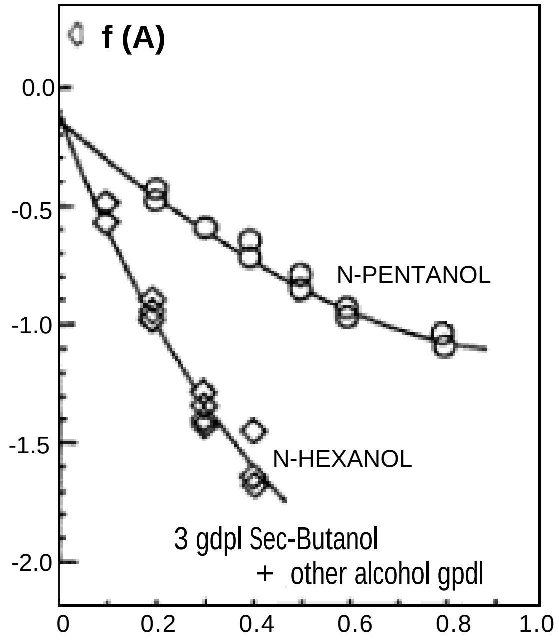

FIG. 10 - fA) FOR 3 gpdl SEC-BUTANOL PLUS VARIOUS CONCENTRATIONS OF n-PENTANOL AND n-HEXANOL

used to characterize the surfactant uniquely, as far as phase behavior and minimum tension are concerned. Fig. 13 shows that EPACNUS correlates linearly with molecular weight for the series of linear alkyl benzene sulfonates.

GEOMETRIC INTERPRETATION

Consider a three-dimensional space, the axis of which corresponds to $X = \ln S^*$, $Y = ACN$, and $Z = f(A)$, and the origin is at (0, 0, 0). In such a space, the correlation for optimal formulation (at 25ºC and WOR = 4) is

$$\ln S^* - K\,(ACN) - f(A) + \sigma = 0,\ .\ . \tag{5}$$

which is the equation of a plane. A normal vector to this plane is (1, - K, 1) and, thus, depends only on K, which has the same value for all sulfonates. As a result, all optimum formulation planes corresponding to different sulfonates are parallel to one another.

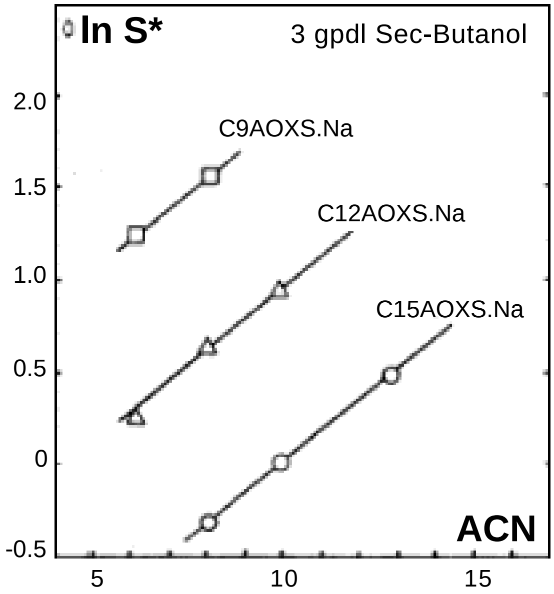

FIG. 11 - CORRELATION ln S* vs ACN FOR THREE ALKYL ORTHOXYLENE SULFONATES OF DIFFERENT HYDROCARBON CHAIN LENGTH.

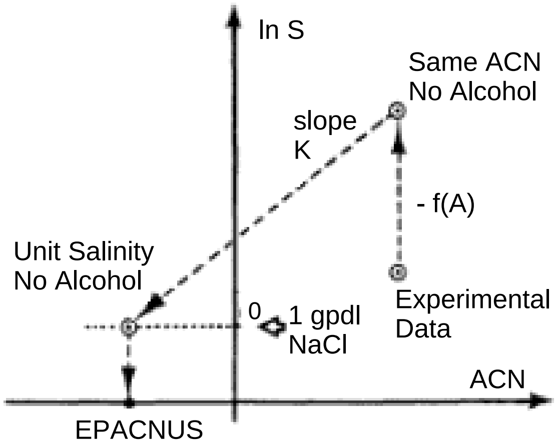

FIG. 12 - GRAPHICAL OBTENSION OF EPACNUS.

Basic analytical geometry can show that the optimal formulation plane cuts the ACN axis at ACN = EPACNUS of the corresponding surfactant. This optimum formulation plane divides the space into two regions. Above the plane,

$$\text{Ln } S^* > K\,(ACN) + f\,(A) - \sigma\ ,\ .\ ,\ .\ . \tag{5a}$$

the system exhibits Type II phase behavior. Below the plane, the inequality (Eq. 5a) is reversed and the system exhibits Type I phase behavior. Finally, on the plane (and near the plane, also, since the optimum formulation is defined as the center of three-phase region), the system exhibits three phases and minimum interfacial tension. This geometric representation allows one to visualize the transition that may be produced by any variable mentioned in Table 1. For instance, a Type I → II transition caused by a salinity increase corresponds to a vertical path crossing the optimum formulation plane from below. A transition resulting from an ACN or an alcohol scan (either concentration or type) would correspond to a path parallel to their respective axis. A transition corresponding to a surfactant molecular weight scan (EPACNUS scan from Fig. 13) would be interpreted as the change of relative position of the point representing the system (ln S*, f(A), and ACN) and the optimum formulation plane that sweeps the space, parallel to itself, when EPACNUS varies.

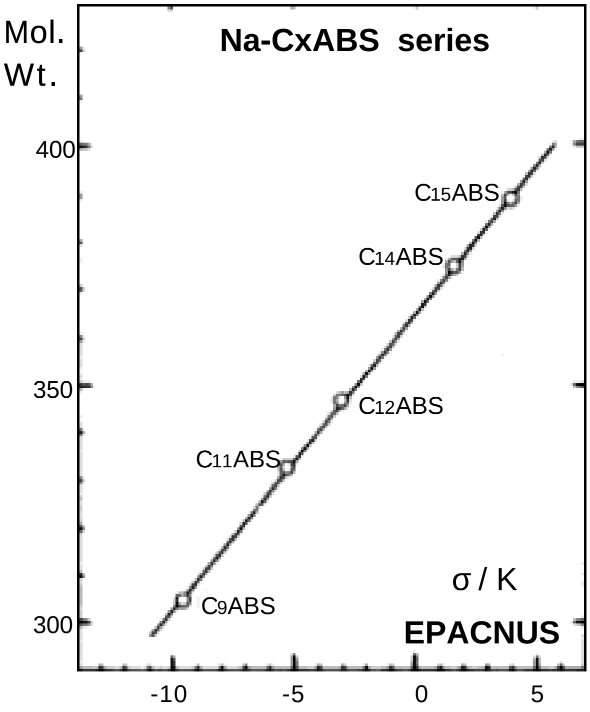

FIG. 13 - EPACNUS VS MOLECULAR WEIGHT FOR ALKYL BENZENE SULFONATE SERIES.

TEMPERATURE AND WOR

The effect of temperature also was studied. Fig. 14 shows the variation of the optimum salinity vs temperature for several systems selected so as to cover a large range of ACN and salinity. Several surfactants - Siponate DS-10, Exxon C_9, C_{12}, and C_{15} oxthoxylene sulfonates, and Witco TRS 10-80 - hydrocarbons ranging from ACN = 6 to 14, and alcohols were used in this study. All data showed the linear relationship indicated by the representative systems plotted in Fig. 14. An average value for the temperature coefficient, a_T, was found to be

$$a_T = \frac{\partial \ln S}{\partial T} \quad \text{(other variable = constant)}$$

$$10^{-2} \pm 0.2 \times 10^{-2} \text{ (ln S units/ºC)} \; \ldots\ldots \qquad (6)$$

This value corroborates the sparse data reported previously [3,17] and is consistent with the value of 0.1 ACN unit/ºC for the low tension (no middle phase) regime reported earlier.[18] The effect of temperature can be included in the correlation, which takes the following form:

$$\ln S^* = K\,(ACN) + f\,(A) - \sigma + a_T\,(T-25) \; \ldots\ldots \qquad (7)$$

As far as the position variables are concerned, we know that surfactant concentration also may affect apparent behavior.[10] The so-called concentration shift also may be seen in Fig. 3; it seems to be related directly to the isomeric purity of the surfactant.

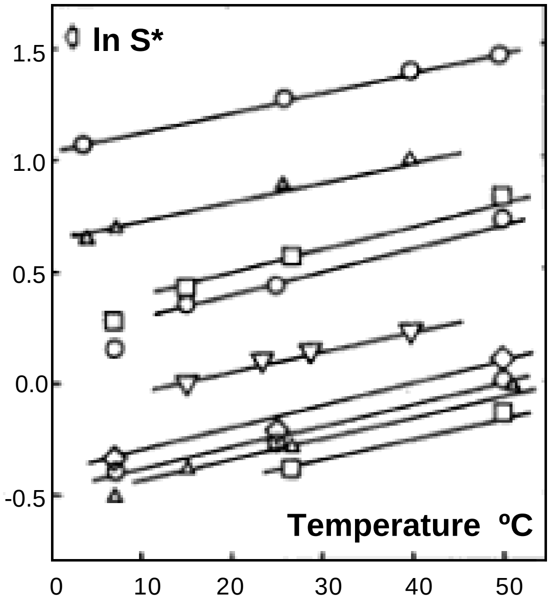

FIG. 14 - VARIATIONS OF ln S* vs TEMPERATURE FOR VARIOUS SYSTEMS (ALKYL ARYL SULFONATES/ OIL/ALCOHOL/BRINE).

Since the position variable effect is limited to the occurrence of Type III diagrams, these are not expected to be very important.[12] Fig. 15 shows that a change in WOR from 4 to I produces only a small change in the position of the correlation straight line, which remains unaffected. Fig. 16 shows the change in preferred ACN vs WOR for several systems; the deviation seems to increase at low WOR, but it may actually result from other phenomena, such as alcohol partitioning.

APPLICATION OF THE CORRELATION

The correlation discussed here applies to alkyl aryl sulfonates, NaCl as electrolyte, and alkanes as the oil. This correlation may be affected by the nature of the surfactant, electrolyte, and oil.[12,13].

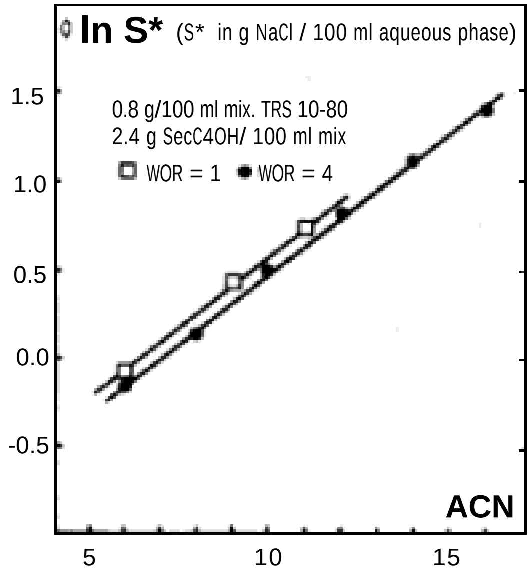

FIG. 15 - INFLUENCE OF WOR ON ln S* VS ACN CORRELATION.

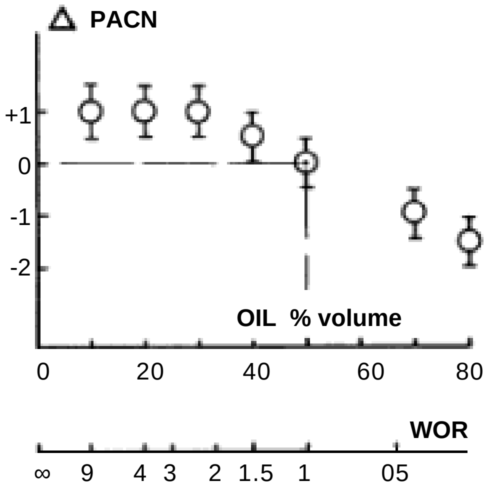

FIG. 16 - SHIFT FROM PREFERRED ACN AT WOR = 1 VS OIL PERCENT VOLUME AND WOR.

Whatever the value of the coefficient K, the use of the correlation is the same: when several variables are determined by external constraints (for instance, salinity and oil ACN), the correlation allows us to estimate the value or values of the adjustable variables (for instance, surfactant structure, alcohol type, and concentration) needed to obtain an optimum formulation. If more than one variable can be adjusted (i.e., if there is more than one degree of freedom), we can select the best optimum formulation according to a supplementary criterion, such as reduced adsorption, minimum tension value, low multiphase region height, or any other practical or economic criterion.

CONCLUSIONS

The optimum formulation of surfactant/water/oil/alcohol systems can be determined either by minimum tension or by three-phase behavior, the two phenomena being essentially equivalent. The optimum formulation is characterized by several concurrent phenomena: (1) minimum interfacial tension, (2) three-phase behavior, (3) high solubilization of both water and oil in the microemulsion phase, and (4) a sharp change of the surfactant partition coefficient, which posses a near unity value for three-phase systems.

A relationship linking the variables that produce an optimum formulation has been proposed for systems containing sulfonates, sodium chloride, water, alkanes, and various alcohols.

For high molecular weight alcohol (or an almost insoluble alcohol), the different variables have separate effects with no cross terms. The main features of the correlation are (1) the logarithm of the optimum salinity varies linearly with oil ACN; (2) for high molecular weight alcohols, the effect of the alcohol can be accounted for through a functional f(A) that depends only on alcohol type and concentration; (3) the surfactant structure is characterized by a parameter σ (σ divided by the slope of the ln S* - ACN correlation is called EPACNUS and is essentially the same as N_{min} defined in previous papers); (4) the temperature effect is linear; (5) the surfactant concentration may have an effect through the so-called concentration shift; and (6) the WOR only affects the optimum formulation slightly.

NOMENCLATURE

a_T = temperature coefficient of optimum salinity expressed in units of ln S per ºC.

ACN = number of carbon atoms in an alkane.

EPACNUS = preferred alkane defined by its ACN of surfactant system at a unit salinity

f(A) = function of alcohol type and concentration.

K = slope of the logarithm of the optimum salinity as a function of ACN.

S = salinity, grams NaCl per 100 cm3 of aqueous phase.

S* = optimum salinity, center of salinity range for which system exhibits three phases.

T = temperature ºC.

γ = interfacial tension.

σ = parameter characteristic of surfactant.

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