

Paper 3

Effect of surfactant structure on foam-oil interactions

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and an alpha olefin sulfonate in static foam tests**

EFFECT OF SURFACTANT STRUCTURE ON FOAM-OIL INTERACTIONS

**Comparing low concentrations of a fluorinated surfactant and
an alpha olefin sulfonate in static foam tests**

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Abstract

Static foam experiments using different low surfactant concentrations of an alpha olefin sulfonate and a fluorinated surfactant have been compared. Both experiments without oil and experiments varying amounts of alkanes as well as crude oil in both synthetic sea water and in 1wt% NaCl solution have been performed. The variation in foam height with surfactant concentration has been compared to changes in bulk aqueous solution with varying surfactant concentration.

Static experiments indicate that foam was generated at a surfactant concentration lower than cmc with oil added. This foam had reduced stability over time.

The fluorinated surfactant (FS-500) gave stable foam at a lower surfactant concentration than the alpha olefin sulfonate surfactant (AOS) in presence of oil. The calculated spreading coefficients of the fluorinated surfactant were negative for all the alkanes at all concentrations. In most cases for the fluorinated surfactant at concentrations 0.01 and 0.5wt% the calculated equilibrium spreading- and entering-coefficients predicted stable foam as observed. No general correlation was found between the foam stability and the spreading- and entering- coefficients for the AOS surfactant. AOS foam tests using alkanes or crude oil were stabilized in low ionic strength solutions.

For both the AOS and FS-500 bulk solutions, speed of sound and viscosity data indicate that changes in the micelle structure occur with increasing surfactant concentration. These changes happened at a concentration close to the surfactant concentration needed to reach a maximum foam height. The change in foam height with surfactant concentration therefore might be associated with the change in the micelle structure from spherical micelles to other more complex structures.

Keywords: Foam, foam-oil interactions, static foam properties, fluorinated surfactant, AOS, low surfactant concentration

Introduction

The stability of foam in the presence of oil is important for applications in oil reservoirs as a gas blocking foam or as foam for mobility control during gas injection [1-3]. However, the factors governing foam stability in the presence of oil are not well understood, and at least four different theories or model have been suggested to describe the system:

1. Spreading and Entering coefficients
2. Lamella number
3. Pseudo-emulsion film theory
4. Bridging coefficient

First, the stability of foam has been related to a negative entering (E) and thus a negative spreading (S) coefficient. These values can be calculated by measuring the surface tension of the aqueous surfactant solution and of the oil in question, in addition to the interfacial tension between the two. Core flood experiments have shown a reduced gas mobility related to systems with a negative entering coefficient thus confirming the correlation between entering coefficient and foam stability [1,4,5]. However, there are also core flood experiments that contradict this correlation [6,7].

The second approach also includes interfacial tensions in defining a Lamella number (L). From dynamic studies in a glass microvisual cell (see reference 1) it was possible to observe if the oil was emulsified and imbibed in the foam or not, and thus correlating lamella numbers to foam stability. Three types of foam were identified, termed A, B and C foams. For type A foams the lamella number is less than 1, for type B foams $1 < L < 7$, and for type C foams $L > 7$. Type A foams are believed to be most stable in the presence of oil. Analyzing this condition in relation to entering and spreading coefficients, it turns out that both are negative for an A foam. Type B foams have a negative spreading coefficient and a positive entering coefficient. These foams are defined to be moderately stability towards oil. Type C foams are unstable foams with positive spreading and entering coefficients.

In other publications the stability of the pseudo-emulsion film has been used to explain foam stability. The pseudo-emulsion film is a thin water layer between the

gas phase and the oil phase. If the pseudo-emulsion film is stable the oil will remain in the lamella. If the pseudo-emulsion film is unstable the oil will form a lens on the surface. This can destabilize the foam. Results in favor of the pseudo-emulsion film theory have been reported [6,8,9].

Also, from studying antifoam agents the water/gas, water/oil and oil/gas, interfacial tensions have been combined to determine what is termed a bridging coefficient (see equation below Table 1). This coefficient has also been used to characterize foams [10-12]. If the oil behaves as an antifoaming agent it is necessary, though not sufficient, that the bridging coefficient is positive. The role of oil-spreading for antifoam activity is a subject of ongoing debate [7,10-12]. A good correlation is often found between antifoaming ability and the positive spreading coefficient, but recent studies have questioned this relationship.

In our previous work [13], foam-oil interactions were first studied for an alpha-olefin sulfonate (AOS) surfactant. Foam was formed at low surfactant concentration, even below the critical micellar concentration, and foam could be stable in the presence of oil. One of the main conclusions, of the earlier studies, was that a negative spreading coefficient was not a necessary condition for stable foam. These experiments also indicated that foam stability in the presence of oil could be related to transport properties within the foam. Further studies were performed comparing an alpha olefin sulfonate and a fluorinated surfactant [14]. The fluorinated surfactant gave stable foams in the presence of oil and performed even better than the AOS surfactant with respect to foam stability.

Most of the earlier experiments using different additives have used a surfactant concentration of 0.5wt%. In this paper, we compare and discuss foam stability and foam – oil interactions at low surfactant concentrations. Experiments with and without different oil additives at low surfactant concentrations are presented. Also, one of the main issues in this study was to investigate properties of bulk solutions of the two surfactants and to try to compare them to the observed variation in foam height.

Materials

The two surfactants used were a C₁₄-C₁₆ alpha-olefin sulfonate surfactant, AOS, and a Perfluoroalkyl betaine, FS-500. These were the same surfactants used in our previous studies [13,14]. The AOS surfactant is anionic with a molecular weight of 324 g/mole and the FS-500 is zwitterionic. According to the vendor the molecular weight of the FS-500 is in the same range as for the AOS. Alkanes used were pentane (Baker, ≥ 99%), octane (Fluka, ≥ 99%), decane (Aldrich, 99+%), and dodecane (Fluka, ≥ 98%). The crude oils, denoted e and f were obtained from the North Sea oil reservoirs. We have used the same notation for the crude oils a –f, to enable the reader to compare to earlier published results [13,14]. The synthetic seawater had the following composition: 2.489wt% NaCl, 0.173wt% CaCl₂, 1.112wt% MgCl₂, 0.019wt% NaHCO₃, 0.406wt% Na₂SO₄, and 0.067wt% KCl; all salts with ≥ 99% purity.

Method

Static foam tests

Foam tests involved static mixing of air into a surfactant solution. The procedure has been described earlier [13]. Air was dispersed into the 300 ml test solution at a speed of 2000 rpm (rounds per minute) for 5 minutes using a pedal connected to a mixer. Solutions were mixed in 1000 ml glass cylinders measuring 44 cm high and 6 cm in diameter. During mixing there was unlimited air supply and no restrictions on foam expansion. After mixing the glass cylinder was closed with a plastic seal. Foam height was always well below the top. In foam tests with oil the amount of added oil was 1wt%, 5wt%, 10wt% or 20wt%, calculated as a weight fraction of the 300 g or 300 ml surfactant solution assuming a density of 1g/ml solution. Oil was added to the surfactant solution before mixing. The oil was therefore dispersed throughout the foam during mixing. In each experiment the height of the foam column above the liquid phase was measured as a function of time after mixing. Both the mixing time and test procedure were identical in each experiment. The experiments were performed at ambient conditions.

The alkanes used were octane, decane and dodecane. Experiments with several combinations of the amount of oil, the alkanes chain length and surfactant

concentration were done for each surfactant. In addition several experiments with FS-500 and 1wt% of pentane were done. All of these experiments were performed in synthetic sea water. The following experiments were done with 1wt% of NaCl solution instead of synthetic sea water: 0.1wt% AOS with 1wt% or 5wt% of alkane; 0.01wt% AOS with 1wt% alkane; and 0.01wt% FS-500 with 1wt%, 5wt% or 10wt% alkane.

Static foam experiments with two different crude oils in synthetic seawater have been performed. This involved investigation of the foam stability using two crude oils where stable foam has been generated earlier with 0.5wt% AOS in synthetic sea water [13]. Experiments were made using oil e and oil f with 0.1wt% AOS and 1wt% of oil. For these oils, static foam tests with 0.1wt% and 0.01wt% FS-500 and 1wt% of oil were also performed.

Viscosity and Speed of Sound of surfactant bulk solutions

In addition to the static foam tests, viscosity and speed of sound measurements were performed for AOS and FS-500 bulk solutions. A large surfactant concentration interval was investigated. The viscosity of the solutions was measured using a Physica MCR 300 Rheometer. All the experiments were performed at room temperature. The shear rate was varied between 10 and 1000 1/s in the measurements, and average values for the viscosity were calculated.

The speed of sound of the bulk surfactant solutions was measured at $(25.000 \pm 0.003)^\circ\text{C}$ by the Rubidium clock sound velocimeter described elsewhere [15]. The speed of sound cells was calibrated with water using the data of Del Grosso and Mader [16]. The estimated error in the measurements was 0.02 m/s.

Surface tension

The interfacial tension of the oils against aqueous surfactant solutions and surface tensions values have been measured by the Pendant Drop method [17]. The instrument used was a CAM 100 with CAM 200 software from KSV Instruments Limited. The surface and interfacial tension of both 0.001wt% and 0.01wt% surfactant against octane, decane and dodecane have been measured for both

surfactants, measuring both initial values and equilibrium values. The surface and interfacial tensions of the different crude oils a-f were measured in our previous studies [13,14].

Results and Discussion

Effect of surfactant concentration

The foam heights for four different surfactant concentrations for the two surfactants are presented in Figure 1. The figure shows the stability of the generated foam in the first five hours after mixing.

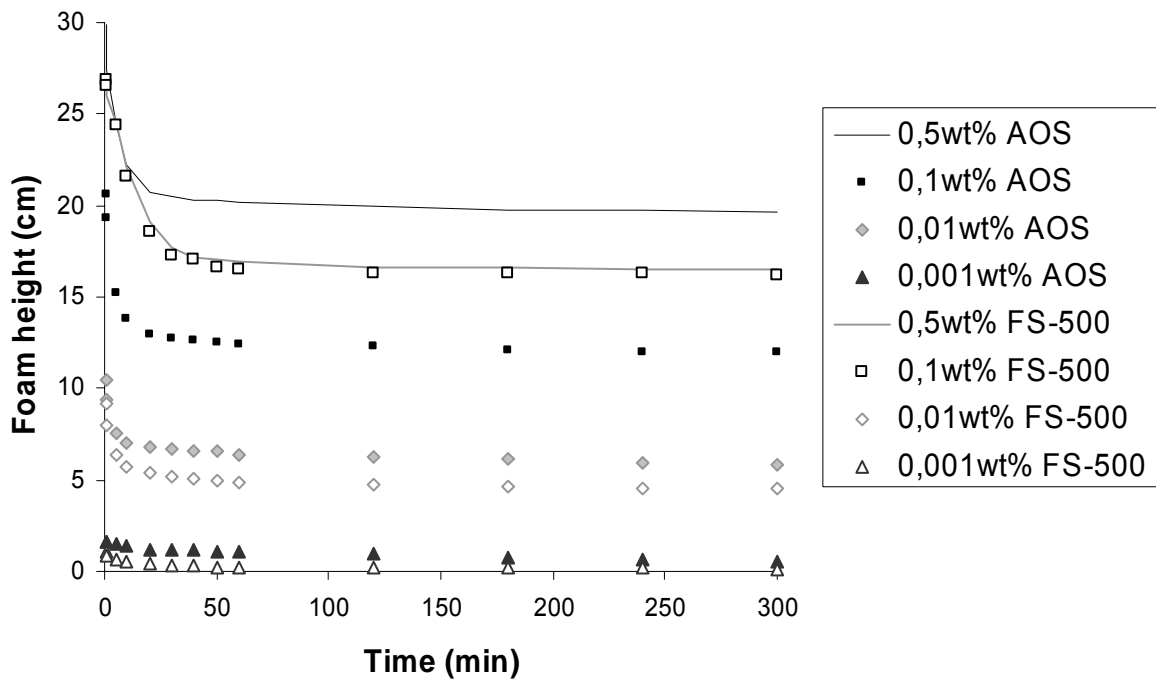


Figure 1: Static foam tests as a function of surfactant concentrations for AOS and FS-500 in synthetic sea water.

The critical micelle concentration (cmc) for the surfactants in synthetic sea water is 0.0022wt% for the AOS surfactant [13] and 0.0028wt% for the FS-500 surfactant [14], measured using the Pendant drop method. As shown in Figure 2 and Figure 3, foam was generated below cmc for both surfactants. Significant amounts of foam are formed below cmc, but the foam heights are low. Using 0.001wt% and 0.01wt% surfactant the foam height was quite similar for both FS-500 and AOS. For 0.1wt%

the FS-500 surfactant generated more foam than the AOS surfactant. No change in foam height was observed when the FS-500 concentration was increased from 0.1 to 0.5wt%.

In our previous paper [13] we investigated the change in foam column height with different surfactant concentrations. The AOS foam reached a maximum height at a surfactant concentration of 0.5wt%. The foam column height increased by increasing surfactant concentration, but did not change when the AOS concentration was increased from 0.01wt% to 0.1wt% (5 x cmc to 50 x cmc, as shown in Figure 2).

As the total surfactant concentration increased from a few times cmc to more than 100 times cmc, the literature suggests that ionic micelles could at some point undergo a transition from near-spherical to rod-like or other elongated forms [18,19]. Others have reported a second cmc at higher concentrations indicating a reorganization of the micelles [20]. For C₁₂-C₁₈ alpha-olefin sulfonate Abed et al. [21] did not observe a transition from spherical to cylindrical micellar shape in the experimental concentration range 0wt% - 3wt%. They used 0-0.5wt% NaCl solution, a significant lower salt concentration than in seawater used in our experiments. Synthetic seawater contains both monovalent and divalent ions.

To investigate this further, the speed of sound and the viscosity of the aqueous surfactant solutions were measured as a function of surfactant concentration both for AOS and FS-500. Figure 2 and Figure 3 show the relative foam height, the relative viscosity and the speed of sound as functions of surfactant concentration, AOS and FS-500 respectively.

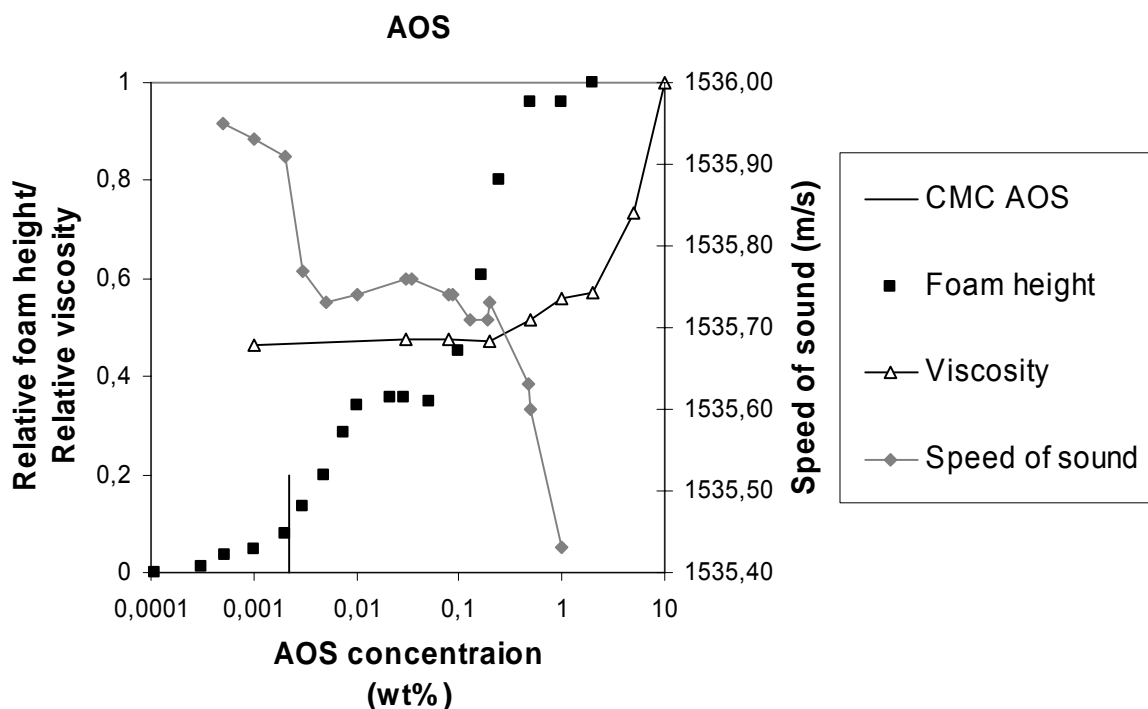


Figure 2: Relative foam height, relative viscosity and speed of sound measurement as a function of AOS concentrations. Foam height was measured 4h after foam generation.

For the AOS system the speed of sound changes surprisingly little with concentration. Nevertheless, two abrupt changes can be identified (Figure 2). The first is a sudden reduction in speed of sound that can be related to the cmc previously measured to 0.0022wt% by surface tension measurements. The fact that the slope becomes more negative indicates a higher compressibility of the micellar state as shown previously [22]. At even higher concentrations there is another sudden decrease in the speed of sound around 0.2wt% AOS. This corresponds reasonably well to the concentration where the viscosity is observed to increase significantly and where the foam height increases. All measurements suggest that changes occur between 0.1 and 1wt% AOS concentration. Abrupt changes in the speed of sound suggest a structural change in the solution, in this case the most probable structural change is from small spherical micelles towards larger ellipsoidal or rod-like micellar shapes. Larger aggregates will also increase the viscosity as observed [23,24]. The two techniques, speed of sound and viscosity is likely to respond differently towards structural changes in solution, and it is thus not surprising that the two points of change do not coincide at exactly equal concentration.

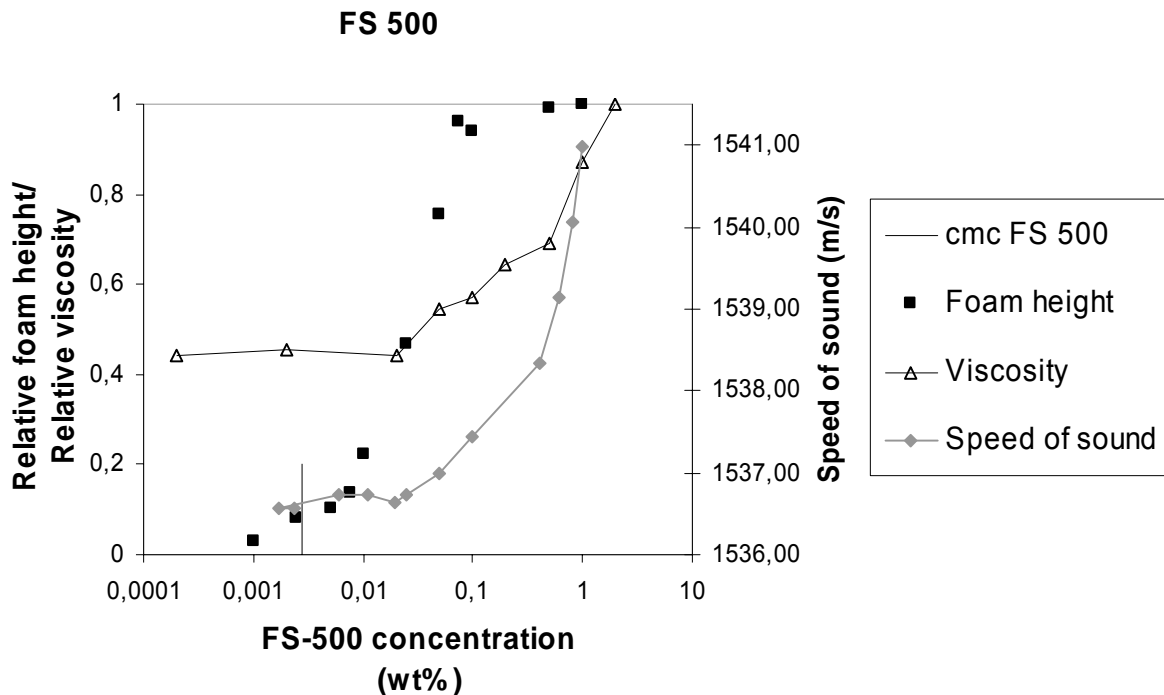


Figure 3: Relative foam height, relative viscosity and speed of sound measurements as a function of FS-500 concentrations. Foam height was measured 4h after foam generation.

Figure 3 shows the FS-500 system. The speed of sound changes significantly more for this system as a function of concentration. Furthermore we observe a significant increase at high surfactant concentration where the AOS system exhibited a decrease. Nevertheless, the most likely conclusion is still a reorganization of the micellar structure around 0.2wt% surfactant concentration. Also for this system the change in speed of sound is reasonably well correlated to an increase in viscosity. Surprisingly it is not easy to detect the cmc by speed of sound measurements. In the AOS system the cmc can be detected both by a sudden change in the speed of sound and by a sudden change in the foam height. For FS-500 neither the foam height nor the speed of sound change significantly at the cmc, 0.0028wt%, as measured by surface tension.

Effect of alkanes

At the lowest surfactant concentration, 0.001wt% surfactant, some foam was generated for many combinations of surfactant and alkane, typical about 1 cm. However, the stability of the foam was poor. After 2-4 hours the foam was broken

down completely, with one exception; in a foam experiment with 1wt% pentane in FS-500 a foam height of 9 cm was observed after four hours. This is consistent with previous studies [13] that have also shown that adding pentane to a FS-500 solution easily generates large amounts of foam.

When increasing the surfactant concentration to 0.01wt%, the observed results for the two surfactants were quite different. (Figure 4; lines are used for illustration). For the AOS surfactant none to less than 1 cm foam was generated in synthetic sea water with any of the alkanes. Replacing the brine with 1wt% NaCl stabilized the foam. In these experiments 3-4 cm of foam was generated. Again, this is consistent with previous studies [13,14]. Less foam was generated in foam tests with oil compared to foam tests without oil for the AOS surfactant.

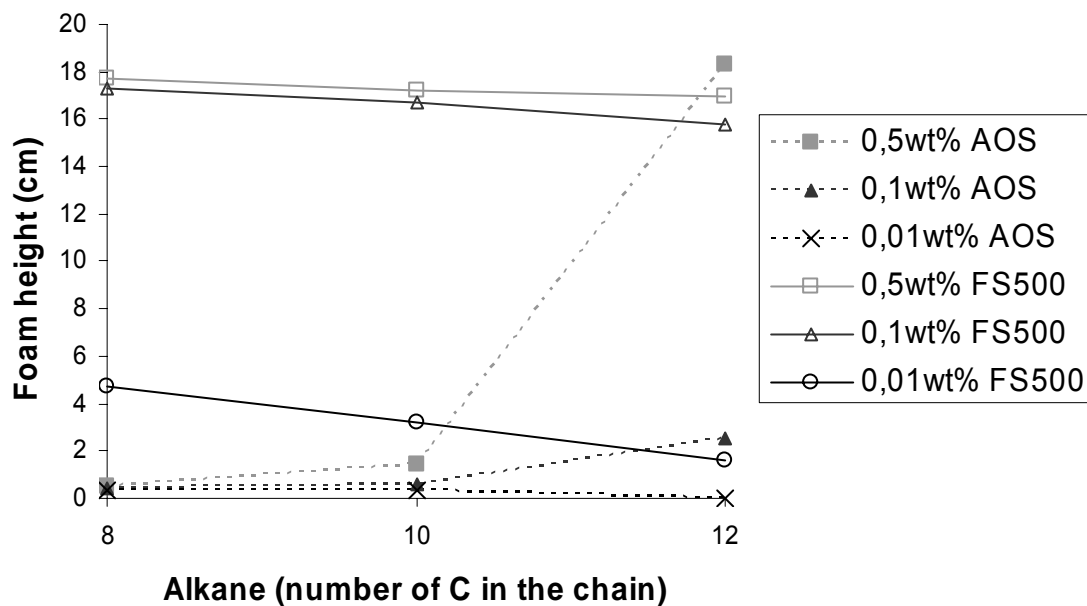


Figure 4: Foam height for 1wt% of different alkanes as a function of number of carbons in the alkane chain. Foam height was measured 4h after foam generation.

For the FS-500 surfactant foam height was 2-5 cm using 1wt% of different alkanes. The foam height increased slightly with decreasing alkane chain length or amount of oil, but any changes were slight. With the addition of 20wt% alkane foam height decreased to 1-2 cm for each of the different alkanes. Changing the salinity of the brine to 1wt% NaCl did not influence the foam height. Foam tests with oil generated equal or slightly less foam than experiments without oil for the fluorinated surfactant.

For the AOS surfactant the same foam height results were observed for 0.1wt% and 0.01wt% surfactant (see Figure 4). The oil clearly reduced foam generation as the foam height was significantly lower for experiments with oil than without oil. The FS-500 surfactant generated 16-18 cm foam for all the foam tests with 0.1wt% surfactant concentration. This was similar to the foam height observed for experiments with 0.5wt% surfactant. It appears that the addition of oil did not reduce foam height at these surfactant concentrations. Foams generated with the fluorinated surfactant FS-500 showed better stability in presence of oil than foams generated with the AOS surfactant. Mannhardt et al. [25] studied three commercial surfactants in bulk foam experiments and observed increased foam stability in the presence of oil with the addition of a fluorinated surfactant.

Spreading-, entering-, bridging-coefficient, and lamella number

Measurements of the interfacial and surface tensions of the surfactants were made and used to calculate the spreading coefficients (S), the entering coefficient (E), the lamella number (L) and the bridging coefficient (B) [1,2,10] at equilibrium conditions for the two surfactants using different alkanes are given in Table 1 (AOS) and Table 2 (FS-500).

At the lowest surfactant concentration, 0.001wt%, both surfactants generated foam in the presence of some alkanes but the stability was poor. The calculated values of S, E, and B for the AOS were consistent with these results. All the corresponding coefficients were large and positive.

The spreading coefficients for the fluorinated surfactant were negative for all the alkanes at all concentrations, see Table 2. In most cases for the fluorinated surfactant at concentrations 0.01 and 0.5wt%, the calculated values of S and E predicted stable foam as observed. In addition the calculated Lamella number - based on studies from Schramm [1] who performed experiments in a micro visual cell (equation given below Table 1)- describes the foams as type A foams believed to have the highest stability in the presence of oil.

The different concentrations of the AOS gave a wide variation in spreading coefficient for the alkanes as concentration was increased from 0.01wt% to 0.5wt%. Observed behavior with respect to foam stability correlated with theory in some cases, but in several cases the observed results contradicted the expected values (Figure 4). The variation in foam stability for the AOS surfactant was neither reflected in the S values nor for the other parameters, E, L and B values.

The stability of the pseudo-emulsion film has not been measured in this study. It is therefore still open that pseudo-emulsions can play an important role in foam stability even for both surfactants.

Table 1: Spreading coefficient (S), Entering coefficient (E), Lamella number (L) and Bridging coefficient (B) at equilibrium for AOS using different alkanes*.

0.001wt%				
Oil	Spreading coefficient (mN/m)	Entering coefficient (mN/m)	Lamella number	Bridging coefficient (mN/m)
Octane	30.0	42	1.5	2916
Decane	30.0	38	2.2	2804
Dodecane	27.6	39	1.5	2786
0.01wt%				
Oil	Spreading coefficient (mN/m)	Entering coefficient (mN/m)	Lamella number	Bridging coefficient (mN/m)
Octane	-0.3	4.7	1.4	106
Decane	-2.7	2.3	1.4	-3
Dodecane	-4.4	1.0	1.3	-77
0.5wt%				
Oil	Spreading coefficient (mN/m)	Entering coefficient (mN/m)	Lamella number	Bridging coefficient (mN/m)
Octane	1.1	5.7	1.6	162
Decane	4.8	6.2	5.4	234
Dodecane	3.0	4.2	5.8	162

*

$$S = \sigma_{w/g} - \sigma_{w/o} - \sigma_{o/g}, \quad E = S + 2\sigma_{o/w},$$

$$L = 0,15 \cdot \frac{\sigma_{w/g}}{\sigma_{w/o}}, \quad B = \sigma_{w/g}^2 + \sigma_{w/o}^2 - \sigma_{o/g}^2$$

$\sigma_{w/g}$: surface tension between water and gas

$\sigma_{w/o}$: interfacial tension between water and oil

$\sigma_{o/g}$: surface tension between oil and gas

Table 2: Spreading coefficient (S), Entering coefficient (E), Lamella number (L) and Bridging coefficient (B) at equilibrium for FS-500 using different alkanes.

0.001wt%

Oil	Spreading coefficient (mN/m)	Entering coefficient (mN/m)	Lamella number	Bridging coefficient (mN/m)
Octane	-9.3	26.1	0.3	747
Decane	-16.3	28.3	0.2	821
Dodecane	-15.2	25.2	0.2	683

0.01wt%

Oil	Spreading coefficient (mN/m)	Entering coefficient (mN/m)	Lamella number	Bridging coefficient (mN/m)
Octane	-8.4	1.4	0.6	-117
Decane	-12.4	1.2	0.4	-191
Dodecane	-13.6	-0.6	0.4	-269

0.5wt%

Oil	Spreading coefficient (mN/m)	Entering coefficient (mN/m)	Lamella number	Bridging coefficient (mN/m)
Octane	-9.2	-2.2	0.7	-203
Decane	-9.7	-1.9	0.6	-192
Dodecane	-15.2	-1.6	0.4	-295

Effect of crude oil

Foam height for 1wt% of oil e and oil f (same terminology as in our previous studies [13,14]) at different surfactant concentrations are given in Figure 5. Foam experiments with 1wt% of crude oil in 0.1wt% AOS in synthetic sea water generated a foam height of only about 1 cm in each experiment. A 1wt% of crude oil in 0.1wt% FS-500 generated stable foam with a height of 16-18 cm in these experiments. This was approximately the same foam height as in experiments with 0.5wt% FS-500, see Figure 5. The same crude oils in foam tests with 0.01wt% FS-500 generated a foam height of 1-3 cm.

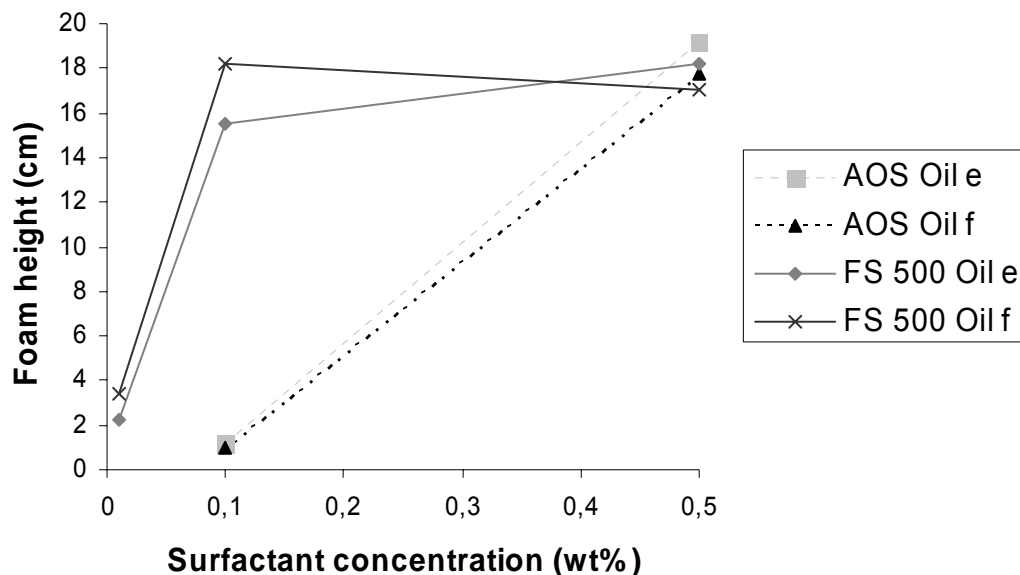


Figure 5: Foam height for two crude oils as a function of surfactant concentrations of the two surfactants in foam tests with 1wt% oil. Foam height was measured 4h after foam generation.

A reduction in the surfactant concentration from 0.5wt% to 0.1wt% did not seem to influence foam height for the FS-500 surfactant. The same changes in surfactant concentration lead to foam breakdown for the AOS surfactant.

Summary

Foam was generated even at a concentration of 0.001wt% for both surfactants, which is at surfactant concentrations 2-3 times lower than the cmc. Both surfactants reached a constant maximum foam height at 0.1-0.5wt% surfactant. Increased concentrations of FS-500 did not show a multiple step increase in foam height. Speed of sound data and viscosity measurements of bulk aqueous solution for both surfactants, suggested changes in the micellar structure with increasing surfactant concentration, and these changes in bulk solutions may be part of the explanation for the observed variation in foam height with surfactant concentration.

In previous studies [13,14] we found that the alkane solubilization was different for the two surfactants. In foam tests with AOS, decane and alkanes with lower molecular weight were solubilized in the micelles. These light alkanes destabilized the foam. The limit for solubilization for the FS-500 was lower. Pentane was solubilized, but alkanes with a higher molecular weight were not. For the FS-500 surfactant, foam tests with pentane gave much more foam than with the other alkanes. A similar result was found in this study. In general foam stability with added oil was poor for both surfactants at a surfactant concentration of 0.001wt%. But, in a foam experiment with 1wt% pentane in FS-500 a foam height of 9 cm was maintained after four hours.

Foams generated with the fluorinated surfactant FS-500 showed better stability in presence of oil than foams generated with the AOS surfactant. For the AOS surfactant a reduced ionic strength increase foam stability in the presence of oil. The most likely argument for this observation of increased foam stability is related to the stability of the pseudo-emulsion film.

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