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G. Morgan[a]; U. Wiesmann[a]
[a] Technical University of Berlin, Institute For Chemical Engineering, Berlin, Germany

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SINGLE AND MULTISTAGE FOAM FRACTIONATION OF RINSE WATER WITH ALKYL ETHOXYLATE SURFACTANTS

G. Morgan and U. Wiesmann*

Technical University of Berlin,
Institute For Chemical Engineering, Sekr. MA 5-7,
Strasse des 17. Juni 135, D-10623 Berlin, Germany

ABSTRACT

The treatment of wastewater containing non-ionic alkyl ethoxylate surfactants with foam fractionation was investigated in the laboratory. The influence of influent concentration, liquid height, foam height, and superficial air velocity on separation characteristics was determined in single-stage continuous operation. Changes in the parameters often had opposing effects on surfactant removal and enrichment. The degree of removal is most dependent upon the ratio of air flow to surfactant load, and the enrichment ratio is most dependent on effluent concentration and foam drainage time. Flotation performed in multistage units had higher removal degrees and lower enrichment ratios than that performed in single-stage units. Up to four columns were operated in series and compared. Through optimal foam heights and superficial air velocities for each stage, the removal degree and the enrichment ratio were

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*Corresponding author. E-mail: gregory.morgan@tu-berlin.de

improved and comparable with single-stage foam fractionation at equal mean residence times, surfactant concentration, and specific air flow rates. Under the chosen experimental conditions, three stages were optimal for separating surfactant from rinse water.

INTRODUCTION

Alkyl ethoxylates (AEOs) are the most widely used group of non-ionic surfactants and represents 55% of the 1.15 million ton, worldwide production of non-ionic surfactants in 1996. In AEOs usage is higher in Western Europe (75%) due to the decision to phase out the use of aromatic polyethoxylated alkylphenols, which have been shown to biodegrade via toxic intermediates (1). Both surfactants investigated, Eumulgin ET5 (ET5) and Genapol UDD-079 (UDD-079), belong to the AEO family but are specialty products for industrial use.

Specialty surfactants are produced in multifunctional batch reactors that must be rinsed between charges. Each rinsing of the reactors, piping, and storage tanks results in large quantities of wastewater with high surfactant concentrations. The reuse of the water from the final rinse in later preliminary rinse processes can reduce the demand for fresh water, but the costs for treatment are not eliminated because the surfactant load is unchanged. In addition, the biological treatment of this wastewater in conventional activated sludge plants is often unsatisfactory and leads to foaming problems, which are combated with anti-foaming agents that further increase the organic load.

An alternative approach to the degradation of the surfactant is the direct treatment of the rinsing waters by physical separation that would allow for the reuse of both water and surfactant. A significant reduction in use of fresh rinsing water is not only ecologically sensible but can also be cost-effective. Foam fractionation employs the characteristics of surfactants to adsorb on the gas-liquid interface of bubbles rising through water to remove them from solution. The foam, which forms at the surface, is allowed to drain and once collapsed, forms a concentrated liquid that could be recycled in the production process.

Conventional flotation makes use of surfactants to improve the separation characteristics during deinking of paper and for separation of minerals, but surfactants themselves are also removed and may become the focal point of a wastewater treatment process in foam fractionation that removes dissolved substances. This process was investigated in the 1970s to alleviate foaming problems in the catching waters of wastewater treatment plants where the recalcitrant surfactants were not removed from the municipal effluents (2). This process still remains interesting for the treatment of industrial wastewater in which specialty surfactants need not adhere to the stringent biodegradability regulations and occur in large quantities. Flotation fractionation may also be useful for the removal of valuable surfactants in permeates resulting from micellar enhanced ultrafiltration processes (MEUF) (3,4).

Several authors have investigated the effects of various parameters on the separation efficiency of surfactants and proteins in batch and single-stage flotation columns, but the use of multistage plants has seldom been reported. The results shown in (4–10) are all consistent in illustrating the relationship between superficial air velocity and removal degrees in single-stage units. Likewise, the strong influence of influent surfactant concentration on the enrichment ratio can be found in (4–7,9). The positive and negative aspects of a multistage process are briefly mentioned in (5), but an optimization of the process and actual experimental results with surfactants alone are not reported.

MATERIALS AND METHODS

Foam fractionation was performed in continuous flow operation with synthetic wastewater consisting of technical-grade surfactants dissolved in deionized water at their natural pH of 6.0 to 6.3. The flotation columns were made of acrylic glass segments with i.d. 7.4 cm and included integrated foam breakers (Fig. 1). The segments could be combined to achieve a height of up to 120 cm.

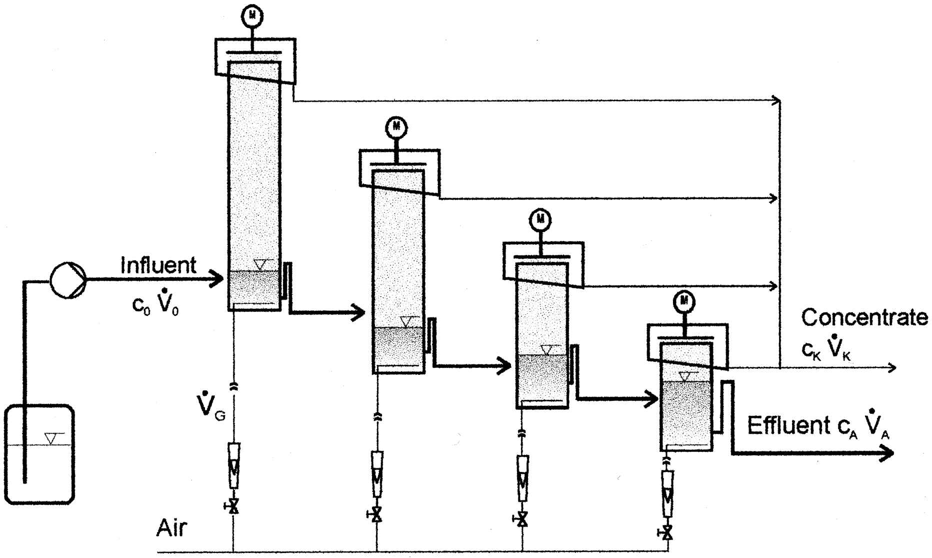

Figure 1. Experimental setup for multistage foam fractionation.

Pressurized air was sparged through sintered glass diffusers of porosity 4 and diameter 5 cm into four units. A description of the investigated surfactants can be found in Table 1. Both AEO surfactants have similar structure (Fig. 2) but have different median hydrophobic and hydrophilic chain lengths. Eumulgin ET5 was provided by Henkel KgaA (Düsseldorf, Germany) and Genapol UDD-079 was provided by Hoechst AG (Frankfurt, Germany) and used without further purification.

Samples were taken and flow rates measured after an operation time equivalent to at least three mean residence times. To ensure that consistent results were obtained during the flotation of ET5, 12 h of vigorous agitation of the influent are required prior to experiments. Surfactant concentration was measured as dissolved organic carbon with a thermo-catalytic method using the DIMATOC-100 from Dimatec (Düsseldorf, Germany). The diffusion rate of the surfactants in water was calculated from the rate of reduction of the dynamic surface tension as a function of the effective surface age as described by (11). The dynamic surface tension of solutions was measured at the surfactant critical micelle concentration (cmc) at 25°C with the maximum bubble pressure method according to (12) using the MPT-2 Tensiometer from Lauda (Lauda, Germany), and the equilibrium surface tension was measured with a Du Nouy ring tensiometer from Krüss (Hamburg, Germany).

Table 1. Non-ionic Alkyl Ethoxylate Surfactants Used

Surfactant Name	Eumulgin ET5	Genapol UDD-079
Alkyl chain length distribution	C14 = 3.4% C16 = 28.3% C18 = 68.3% (C18 includes singly and doubly unsaturated compounds) *	C9 25% C10 44% C11 31% *
Mean degree of ethoxylation	*m* = 5 *	*m* = 6 *
HLB***	9	13
Mol mass	475 g/mol	390 g/mol
Critical micelle concentration (cmc) and surface tension	17.5 mg/L σ_{cmc} = 30.6 mN/m (25°C)	250 mg/L ** σ_{cmc} = 26.7 mN/m (25°C) **
Diffusion coefficient	D = 3.61×10^{-8} cm^2/s	D = 1.26×10^{-6} cm^2/s

* (16) ** (17) *** HLB = hydrophilic-lipophilic balance

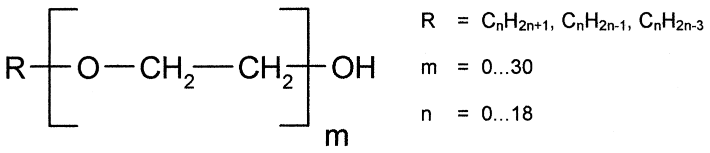

Figure 2. Structure of alkyl ethoxylates. ET5: $n = 14–18$; $m_{\text{average}} = 5$; UDD-079: $n = 9–11$; $m_{\text{average}} = 6$.

RESULTS AND DISCUSSION

The influences of several parameters were investigated in a single-stage operation. This process was evaluated by removal degree α and enrichment ratio β, which should both be maximized.

$$\alpha = \frac{c_0 - c_A}{c_0} \qquad \beta = \frac{c_K}{c_0}$$

The surfactant concentrations are designated c_0 in the influent stream, c_A in the effluent stream, and c_K in the foam concentrate stream.

While an increase in the mean residence time $t_V = V/\dot{V}_0$ improves both the removal degree and the enrichment ratio, other factors that could influence the performance of the system need to be optimized. The heights of the water and the foam, the air flow rate, and the influent surfactant load were varied independently in the column.

Influence of Water Height

Variation of the water height in the column during the foam fractionation of ET5 rinse water showed a minimum height necessary to ensure maximum removal at surfactant load and superficial air velocity (Fig. 3). At water heights below $H_L = 6$ cm, the bubbles were evidently not fully saturated with surfactant monomers and the removal degree α was lower. An increase in H_L beyond 6 cm at maintained influent concentration increased the volume of the column, and thus also the mean residence time, but led to no increase in α. Water heights below $H_L = 6$ cm led to an increase in β. This is due to the lower stability of the foam caused by insufficient surfactant adsorption. Bubbles with lower surfactant concentrations at the boundary layer are less stable and carry less water in the foam films. Lower surface concentration may yield lower surface viscosity, which increases the rate of drainage. A lower water content results in less dilution of the adsorbed surfactant molecules and thus a higher enrichment ratio. The difference between these results and the results from (4), in which a positive relationship between liq-

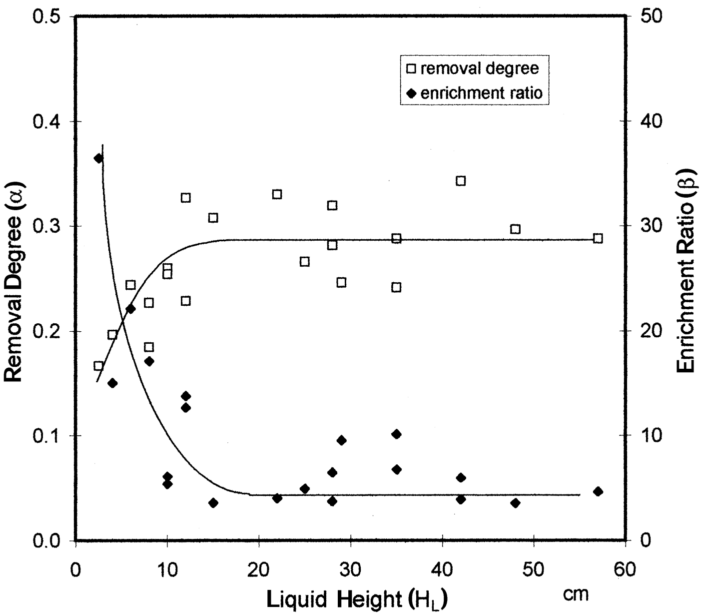

Figure 3. Influence of liquid height on removal degree and enrichment ratio; $c_0 = 500$ mg/L ET5; $\dot{V}_L = 560$ mL/h; $\dot{V}_G = 15$ L/h; $H_F = 23$ cm.

uid height and enrichment ratio was shown, can be attributed to the differences in surfactant type and range of liquid heights investigated. Therapiwattananon et al. (4) limited the minimum liquid height to 15 cm, which may not have been sufficient to identify the effects described here. The anionic surfactants used produce much more foam at concentrations well below the cmc, where the time needed for complete adsorption at the interface may be several hundred milliseconds. The greater porosity of the sparger system used by (4) also led to larger bubbles with higher rising velocities than those found in this study.

Influence of Foam Height

Variation of the foam height H_F has a more direct effect on the water content of the foam than does water height. An increase in H_F leads to longer foam residence times, which allow for more drainage of the liquid in the films. The dilution of the adsorbed surfactant molecules is lower when H_F values are high, which in turn leads to higher enrichment ratios β (Fig. 4). Within the range of foam heights investigated here, no significant effect on α was found. These results are in agreement with those from Tharapiwattananon et al. (4).

Influence of Superficial Air Velocity

The influence of the superficial air velocity $w_G = \dot{V}_G/A$ on the separation characteristics was also investigated (Fig. 5), and the results were similar to those of other researchers (4,5–10). With both surfactants, an increase in w_G values were consistent with a rise in α values, which plateau, as well as a significant reduction in β. An increase in bubble production with rising w_G leads to a greater surfactant mass transport out of the column with the larger surface area for adsorption, but the foam drainage is limited by the shorter foam residence time. As a result of different adsorption rates, a significant difference between the two surfactants was found in the removal degree attained by foam fractionation.

Influence of Influent Concentration

The influence of the influent concentration c_0 on the foam structure is very dramatic. Similar results were found for both surfactants by varying c_0 (Fig. 6). At the lowest ET5 influent concentration of $c_0 = 375$ mg/L and a superficial air velocity of $w_G = 0.15$ cm/s, effluent concentrations of $c_A = 298$ mg/L and $c_K = 134{,}000$ mg/L were reached, giving the highest value of $\beta = 358$. A very similar

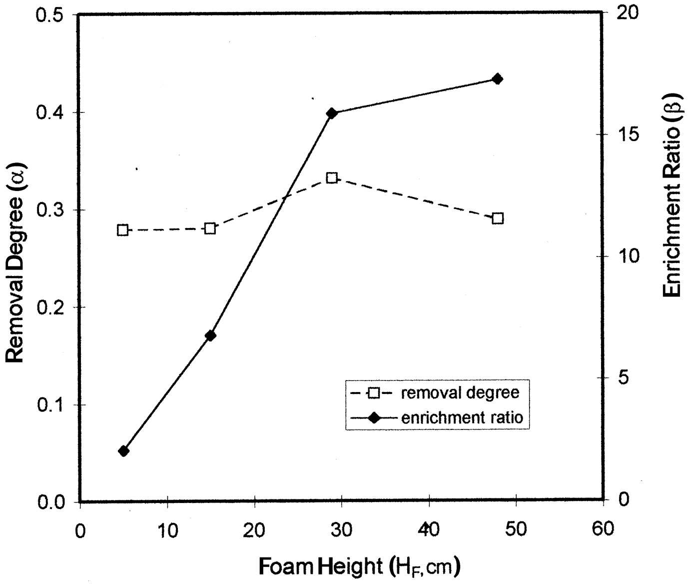

Figure 4. Influence of foam layer height on removal degree and enrichment ratio. $c_0 = 500$ mg/L ET5; $\dot{V}_L = 560$ mL/h ; $\dot{V}_G = 15$ L/h; $H_L = 12$ cm.

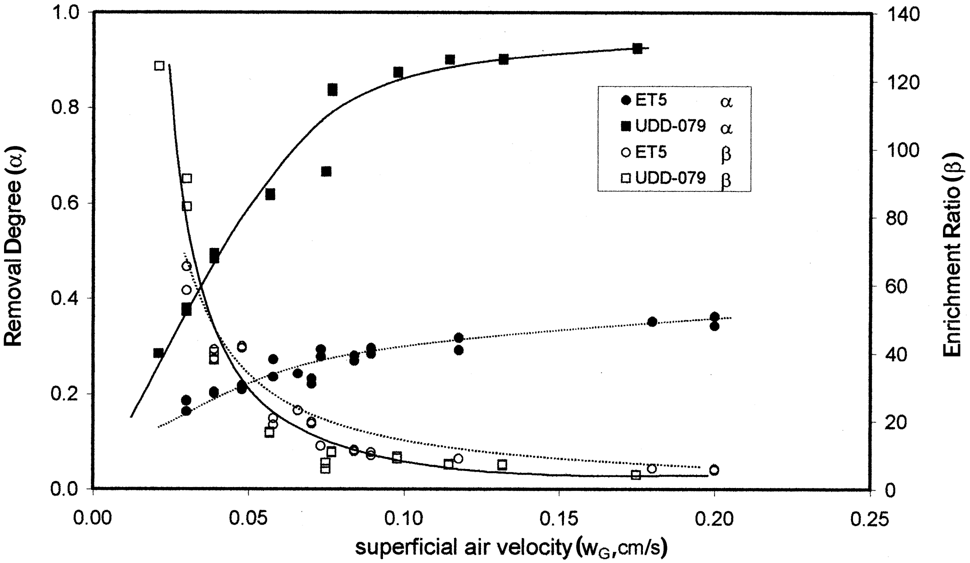

Figure 5. Influence of the superficial air velocity w_G on the removal degree α and enrichment ratio β: ET5 and UDD-079 at identical conditions: $c_0 = 500$ mg/L, $\dot{V}_L = 560$ mL/h; $H_L = 10$ cm; $H_F = 42$ cm.

relationship between c_A and c_K was also observed with UDD-079 but at a lower influent concentration range. While still reaching a high concentration values, UDD-079 concentrations are much lower than those of ET5 at equal residence time and air flow rate. At the lowest UDD-079, the influent concentration c_0 = 100 mg/L, c_K = 36,840 mg/L, c_A = 7.1 mg/L, α = 0.93, and the highest enrichment was reached: β = 362. Figure 7 shows the relationship of the effluent to the collapsed foam for both surfactants at the different influent loads. The inverse relationship between c_A and c_K results from the stability characteristics of the foam, which depend on the surface concentration and surface viscosity. Foam that is formed over a fluid with low surfactant concentration is less stable and results in a much higher enrichment ratio than that formed over high-surfactant fluid. The foam formed over a fluid with higher concentration is characterized by smaller, more stable bubbles. The surface area for the adsorption of surfactant monomers is greater, but the water content of the foam is consequently much higher. The sharp rise in c_K at c_A values below UDD-079 quantities of 50 mg/L signal the best conditions for the use of foam fractionation to retrieve surfactants from rinse water. Similar results were observed by Wungrattanasopon et al. (9).

As evident from the experimental results, to achieve high enrichment values, foam fractionation is best used at lower surfactant concentrations, such as for

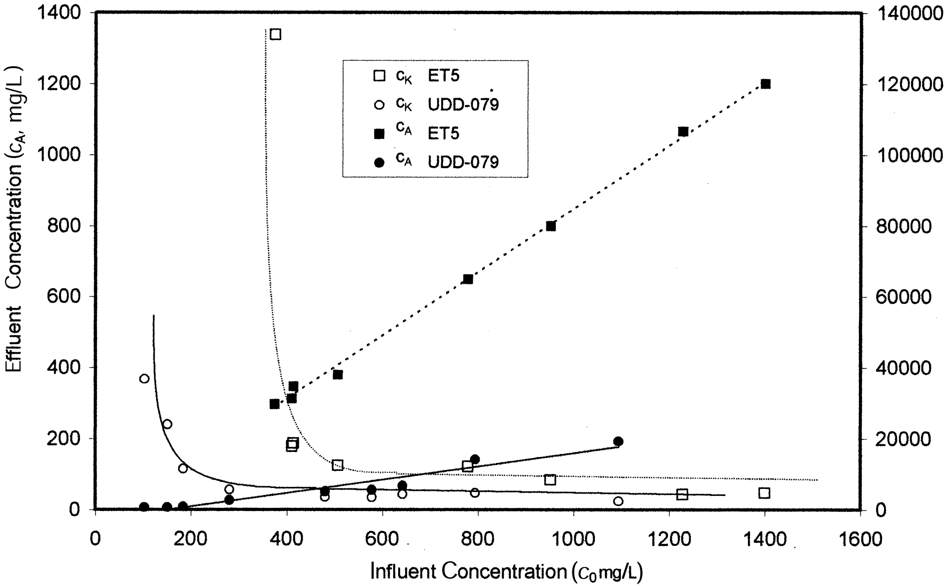

Figure 6. Influence of influent concentration on foam and effluent concentrations: ET5 $c_0 = 270\text{–}1000$ mg/L^{-1}; UDD-079 $c_0 = 100\text{–}1000$ mg/L $\dot{V}_L = 560$ mL/h; $\dot{V}_G = 10$ L/h; $H_L = 10$ cm; $H_F = 42$ cm.

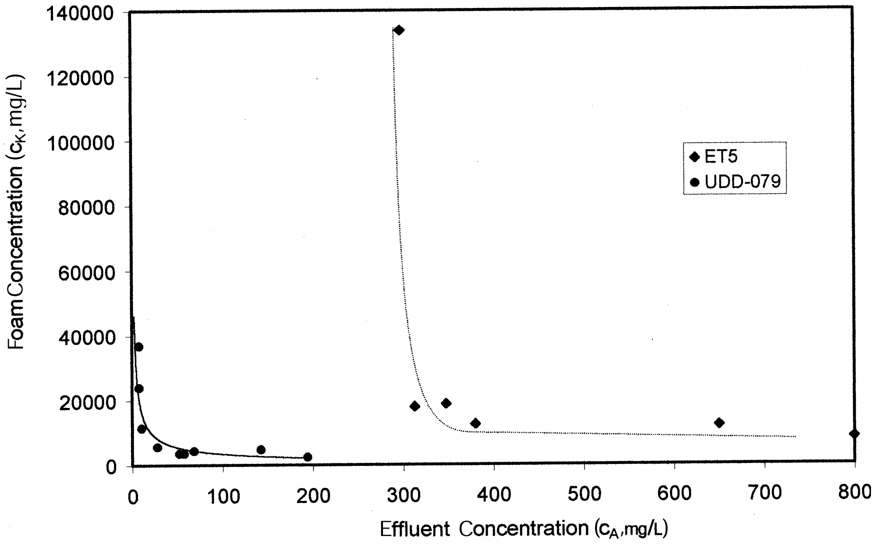

Figure 7. Relationship between effluent and concentrate, parameters as in Figure 6.

the permeates from ultrafiltration of rinse water (13) or MEUF of wastewater, where micelles do not pass the membrane and permeate concentrations lie at the cmc (14). Typical concentrations of 5–20 g/L surfactant have been measured in the enriched stream from foam fractionation and could be fed into a membrane separation unit.

Single and Multistage Foam Fractionation of ET5

The typical removal degree of $\alpha = 0.30-0.50$ in a single-stage foam flotation unit at residence times near $t_V = 3$ h for ET5 was not satisfactory. The total removal degree must be increased by further treatment of the effluent in subsequent stages. However, an increase in α as a result of the multistage treatment system should not be solely dependent on an increase in t_V. Although α was nearly constant with respect to c_0, one can see from Fig. 8 that the absolute concentration reduction from influent to effluent rises sharply with influent concentration. Therefore, use of multistage processes is advantageous for reaching lower effluent concentrations where all but the last of the partial volumes of the system are at higher concentrations, yielding greater total removal. For the purpose of comparison, a single-stage and a two-stage setup were operated with identical residence times, influent concentrations, and specific air flow rates. The cascading of

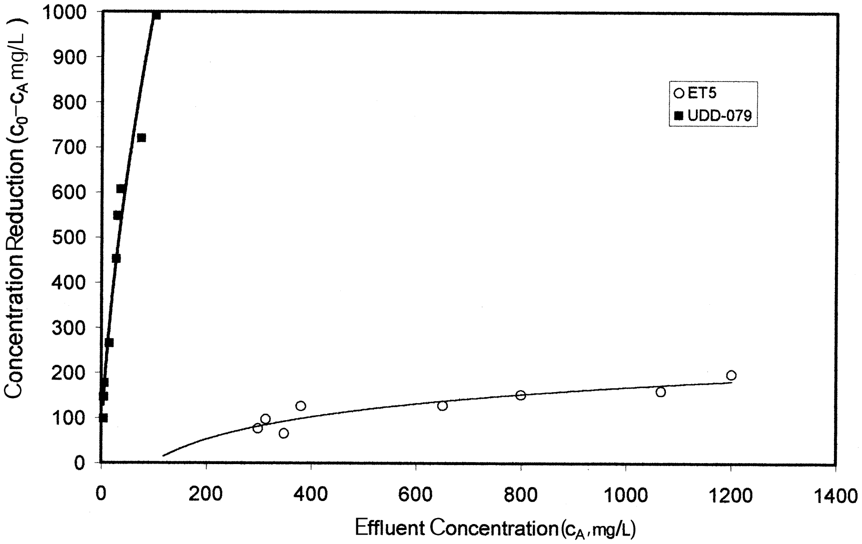

Figure 8. Concentration reduction dependent upon effluent concentration, parameters as in Figure 6.

Table 2. Comparison Between One- and Two-Stage Foam Fractionation of ET5 at Identical Specific Air Flow Rate and Residence Time[a]

Experimental Results		One Stage	Two Stages
Effluent stage 1	c_{A1}	209 mg/L	241 mg/L
Removal degree stage 1	α_1	0.375	0.280
Effluent stage 2	c_{A2}		182 mg/L
Total removal degree	α_Σ	0.375	0.455
Enrichment ratio stage 1	β_1	60.0	12.2
Enrichment ratio stage 2	β_2		30.2
Enrichment ratio for mixed concentrate streams 1 & 2	β_Σ		15.8

[a] $c_0 = 500$ mg/L ET5; $\dot{V}_L = 560$ mL/h; $w_G = 0.097$ cm/s; $t_V = 1.5$ h.

two flotation columns led to an increase in the removal degree from $\alpha_1 = 0.375$ to $\alpha_\Sigma = 0.455$ (Table 2). The enrichment ratio in the two-stage setup was significantly lower than in the single-stage flotation unit. Although the concentrate of the second stage was highly enriched, the enrichment value for stage 1 was too low. Considering the results shown in Fig. 7, it is to be expected that the foam generated in a stage with a higher effluent concentration, as in stage 1, has a lower concentration. Mixing both concentrate streams together resulted in a much lower β value than that obtained from the single-stage method. The concentrate flow rate from stage 1 is four times as high as that from stage 2 and influences the average concentration much more than did the stage 2 component.

Both the relationship between concentration reduction ($c_0 - c_A$) and effluent concentration c_A, as well as the relationship between c_K and c_A, should be considered in the choice of a single or multistage mode of surfactant removal. A single-stage treatment unit operates at the desired low-effluent concentration, but the degree of surfactant removal degree is low and a longer t_V is needed. However, the enrichment in a single-stage unit is much greater than in the two-step model. A multistage unit has partial volumes of higher concentrations and the total removal degree is higher, but the mixed concentrate streams have lower enrichment. The importance of the parameters α and β will need to be assessed when a decision about operation methods must be made.

Multistage Foam Fractionation of UDD-079

Compared to ET5, UDD-079 can be removed at much lower effluent concentrations by foam fractionation because of its higher affinity to the boundary layer. Although it belongs to the same class of surfactants, the length of its hydrophobic chain and number of hydrophilic units differ from that of ET5, giving it a much higher cmc, greater diffusion coefficient, and lower foam limit (under 5 mg/L). As is true for ET5, the foam fractionation of UDD-079 at higher influent concentrations led to greater surfactant removal but also a high water content of the foam. Multistage foam fractionation units can be advantageous for reducing surfactant concentration from high levels to a minimum value. As dictated by adsorption equilibrium, greater adsorption of monomers at higher local surfactant concentrations will be observed at each stage and greater removal with foam bubbles will be achieved. However, the effects of multistage operation on the enrichment ratio must also be considered.

The influence of the superficial air velocity on the removal degree and concentration factor for different influent concentrations between 100 and 500 mg/L is shown in Fig. 9. The values for α_i and β_i are defined with respect to the nominal influent concentration of 500 mg/L; that is, $\alpha_i = (c_{0i} - c_{Ai})/500$ and $\beta_i = c_{Ki}/500$. The greatest reduction in concentration from influent to effluent was found at the highest influent concentration, where the effluent concentration and surface excess in the foam are the highest. At higher effluent concentrations, the lower surface tension of the solution stabilizes smaller bubbles, yielding a larger total surface area for adsorption and generating a higher degree of removal. The resulting higher c_A influences the shifting of the point of inflection for the curves

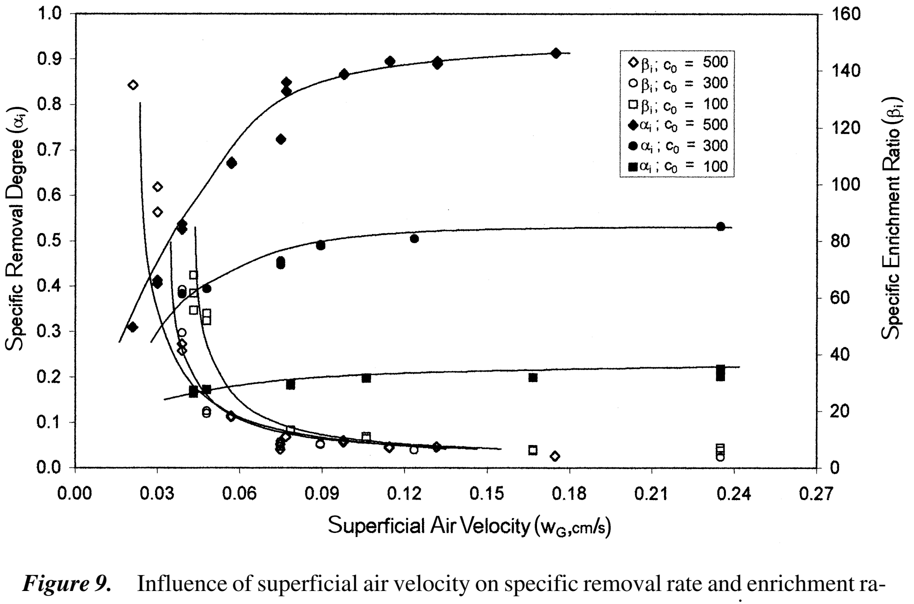

Figure 9. Influence of superficial air velocity on specific removal rate and enrichment ratio at different influent concentrations of UDD-079. $c_0 = 100$–500 mg/L; $\dot{V}_G = 560$ mL/h; $H_L = 10$ cm; $H_F = 42$ cm.

Table 3. Process Parameters During Four-Stage Foam Fractionation of UDD-079

Influent concentration c_0 (mg/L)	500			
Influent flow rate $\dot{V}_0$ (L/h)	1.14			
Mean residence time t_V (min.)	120			
	Stage 1	Stage 2	Stage 3	Stage 4
Liquid height H_L (cm)	10	13	14	15
Foam height H_F (cm)	50	41	24	14
Air flow rate $\dot{V}_G$ (L/h)	5	10	15	30

that describe the influence of w_G on β_i. With increasing c_0, lower w_G values are necessary to achieve high levels of enrichment.

A lower α_i will result from the choice to lower w_G to increase the enrichment in a unit operating at higher influent concentrations. However, α_i remains higher than in a unit at lower influent (and effluent) concentrations (see Fig. 9; $w_G = 0.02$ cm/s for $c_0 = 500$ vs. $c_0 = 100$ mg/L). Therefore, surfactant removal and greater enrichment in the initial stages of a multistage unit can be greater than those in a single-stage unit operating at the desired final effluent concentrations. At a given residence time, specific air flow rate, and by controlling the dosage of air to the stages, higher concentrations and lower effluent concentrations than in the single-stage system can be achieved in a multistage system.

Experimental verification of this premise was performed in foam fractionation units of one, two, three, and four stages operating at an equal influent concentration $c_0 = 500$ mg/L, specific air flow rate $Q = 118$ g air/g surfactant, and mean residence time of $t_V = 2$ h. In all multistage experiments, the lowest superficial air velocity was chosen for stage 1, was increased from stage to stage, but had the same (total specific air)/(surfactant load) as in the single stage unit (Table 3). The height of the foam layer was also reduced from stage to stage to provide adequate foam drainage time in the first stages and to ensure that the foam reached the breaker in the last stage.

Compared to the single-stage unit, the two-, three-, and four-stage units yielded lower effluent concentrations (Table 4). By dividing the unequal amount of air that was going to the different stages, a high enrichment ratio, $\beta_\Sigma > 10$, was maintained for the mixed concentrate streams, which was comparable to that found from the single-stage unit, and the degree of separation, relative to the single-stage operation, was increased. Figure 10 shows the concentrations of the streams within the three-stage unit. The fall in c_K from stage to stage does not follow the relationship found in Fig. 7, as the combined influences of the superficial air velocity and foam height overlap the positive influence of lower c_A on c_K.

Table 4. Variation of Number of Stages[a]

			(Mixed Streams)	
No. of Stages	c_A (mg/L)	α	$c_{K\Sigma}$ (mg/L^{-1})	β_Σ
1	59.2	0.877	2590	10.9
2	37.3	0.923	6269	12.7
3	20.2	0.959	6384	13.4
4	13.7	0.972	2950	6.1

[a] $Q = 118$ g air/g UDD-079; $c_0 = 500$ mg/L; $t_V = 2$ h.

The optimum number of stages under these conditions is three, as is evident by the near- maximum value of $\alpha = 0.959$ and highest value for $\beta_\Sigma = 13.4$. A higher number of stages led to a greater water content in the foam of the first stages under higher effluent concentrations but not to a significant improvement in the removal degree. The increase in the level of complexity with more than three stages is not justified. This nonlinear behavior results from the inverse relationship between c_A and c_K.

The multistage mode of operation with concentrated streams being gathered from each stage of the unit would appear to be less effective than, for instance,

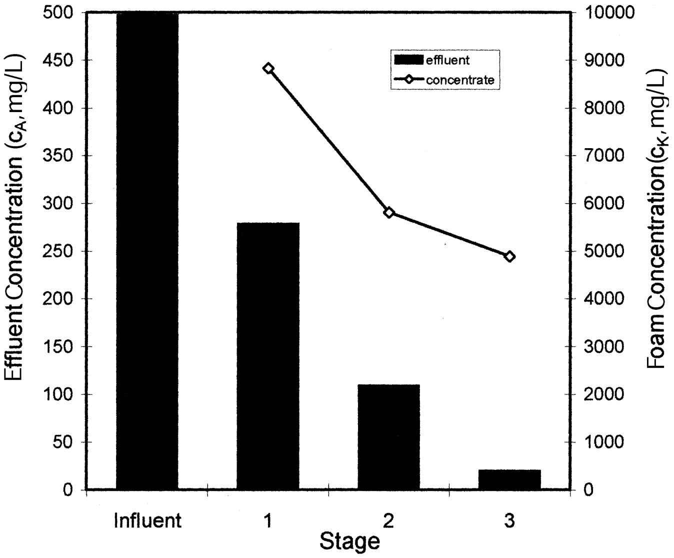

Figure 10. Four-stage foam fractionation of UDD-079. c_0=500 mg/L; t_V = 2h; Q = 118 g air/ g UDD-079; other parameters described in Table 3.

countercurrent operation, as suggested by (15), which is analogous to the distillation of mixtures. However, countercurrent operation did not lead to an improvement in the separation characteristics of multistage foam fractionation units. The relationship between the two concentrations c_A and c_K (Fig. 7) shows that the quasi-equilibrium between the two effluent streams is inverse. In spite of the falling surface concentration, Γ , as bulk concentrations fall below the cmc, as described by adsorption equilibrium, the highest values of c_K occur in the concentrate formed in the stage with the lowest c_A; the effluent concentration was well below the cmc of UDD-079. The addition of the lower-stage concentrate to the next higher stage would raise the surfactant concentration in the higher stage and result in poor drainage of the foam and thus lower surfactant concentration in the higher-stage concentrate. In the case of foam fractionation of surfactants, the separation efficiency would not be improved by countercurrent flow.

SUMMARY

The foam fractionation of rinse water containing surfactants was investigated in a continuous flow operation. The parameters of influent concentration, air flow rate, liquid and foam heights were investigated in single-stage operation for two alkyl ethoxylate surfactants. High values of $\alpha = 0.93$ and $\beta = 362$ could be obtained for UDD-079, which is characterized by a higher diffusion coefficient and higher cmc than those of ET5. The removal degree was improved in the multistage operation. An inverse relationship between the surfactant concentration in the effluent and concentrate streams was identified. This situation led to an overall worsening of the concentration factor in multistage operation, but the use of optimized specific air velocities and foam heights in each stage resulted in an improvement in both removal degree and enrichment ratio. In multistage operation, the removal degree and the enrichment factor for the surfactant load investigated was optimal when number of stages, n, was equal to 3.

NOMENCLATURE

A	Cross-sectional area of column (cm^2)
c_0	Concentration in influent (mg/L)
c_A	Concentration in effluent (mg/L)
c_K	Concentration in collapsed foam (mg/L)
D	Diffusion coefficient (cm^2/s)
H_F	Foam height (cm)
H_L	Liquid height (cm)
n	Number of stages

Q	Ratio of air mass flow to surfactant influent mass flow
t_V	Mean residence time (h)
V	Liquid pool volume (L)
$\dot{V}_0$	influent flow rate (mL/h)
$\dot{V}_A$	effluent flow rate (mL/h)
$\dot{V}_K$	concentrate flow rate (mL/h)
w_G	Specific air velocity (cm/s)
Γ	Surface concentration (g/cm^2)
α	Removal degree
α_i	Specific removal degree relative to nominal influent
β	Enrichment ratio
β_i	Enrichment ratio relative to nominal influent
β_Σ	Enrichment ratio for mixture of all concentrate streams

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