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## Surfactant Recovery from Water Using a Multistage Foam Fractionator: Effect of Surfactant Type

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**Abstract:** The purpose of this study was to investigate the recovery of surfactants using a multistage foam fractionator for three types of surfactants: cationic (cetyl pyridinium chloride, CPC); anionic (sodium dodecyl sulfate, SDS); and nonionic (polyoxyethylene(20) sorbitan monolaurate, Span80). The studied system was operated at a constant temperature of 25°C with a surfactant concentration in the range of 50 to 100% of CMC (critical micelle concentration). For any surfactant system, the enrichment ratio of surfactant increased with increasing foam height and number of stages but decreased with increasing effects of the air flow rate and feed concentration. For all studied surfactants, the removal efficiency of the surfactant was not significantly affected by changing the air flow rate, foam height, and feed concentration in the studied ranges. An increase in the number of stages showed a great improvement of both the enrichment ratio and the removal fraction for all three types of surfactants. In a comparison among the three studied surfactants, the separation performance, in terms of the enrichment ratio and removal was found to lie in the following order: CPC >Span80 > SDS, which can be explained by the foamability and foam stability of each surfactant.

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**Keywords:** Foam fractionation, surfactant recovery

## INTRODUCTION

Surfactants can be present in various industrial wastewaters at different concentrations. The surfactants must be reduced in concentration in order to meet the environmental standards before the discharging of these wastewaters to the environment. Foam fractionation is a surfactant-based separation process that can remove pollutants from wastewater and groundwater (1–3). In addition to satisfying environmental regulations, the value of the surfactant being emitted sometimes makes recovery operations more economical. An alternative approach to the biodegradation of surfactant-containing wastewaters is the direct surfactant recovery by physical separation that would allow for the reuse of both the water and the surfactants. Several wastewaters, which typically contain very low surfactant concentrations around or below the critical micelle concentration (CMC), can possibly be treated to separate surfactants economically by using the foam fractionation technique, as demonstrated by our previous work (4).

Foam fractionation is one member of a group of processes known as adsorptive bubble separation techniques, which isolate species based on surface activity (5). Foam fractionation processes have been used to concentrate and remove surface-active agents from aqueous solutions (6). Foam fractionation is based on the selective adsorption of solutes at the gas-liquid interface, which is generated by a rising ensemble of bubbles through the solution. This ensemble of bubbles forms a foam bed (on top of the liquid pool) which preferentially contains the surface-active solutes (7). The water contained in the foam naturally drains off due to gravitational force and the foam eventually collapses to form a concentrated liquid that can be recycled in the production process. Foam fractionation as a separation technique for homogeneous liquid mixtures has high efficiency at low surfactant concentrations, unlike many conventional methods of separation. To achieve the separation in the foam fractionation operation, adequate foamability and foam stability are required to ensure that the generated foam reaches the outlet at the top of the column.

There are two operation modes of foam fractionation: batch and continuous modes (8). The foam fractionation column can also be classified into two categories: single-stage and multi-stage. Most studies have used both batch and continuous modes in single-stage foam fractionation columns (5,6,8–14). To evaluate the performance of foam fractionation units, many operational parameters are considered to affect the surfactant

removal efficiency, including feed concentration, air flow rate, bubble size, and foam height. Other important parameters are sparger geometry, which has to be suitable for the operational design of the column such as the frit or small porosity, height of foam-liquid interface, and flow pattern direction (15,16). The insertion of perforated plates was found to enhance the enrichment of the surface active material in a multistage foam fractionation unit. A mathematical model of a multi-staged foam fractionation system using the Langmuir adsorption isotherm and a mass balance of each stage in the column was developed and verified experimentally for two types of surfactants: octyl-phenol polyethoxylate (OPEO<sub>10</sub>) and cetyl pyridinium chloride (CPC) (17). The use of perforated plates in a foam fractionation column with external reflux was found to reduce the liquid holdup in foam, resulting in an increase in the enrichment ratio of poly(vinyl alcohol) (18).

Foam characteristics play an important role in governing the surfactant recovery performance, apart from operational parameters such as feed flow rate, surfactant concentration, air flow rate, foam height, and number of stages. The chain length of the hydrophobic part of the surfactants plays a determining role with respect to the ensuring surface activity and foam stability (19). Foam stability also depends on surfactant concentration of the foaming solution. At higher temperatures (typically >35°C), the coalescence of foam becomes dominant, resulting in lowering of the foam stability (20).

The objective of this study was to compare the process performance of a multistage foam fractionation system for recovering three different types of surfactants: cationic, anionic, and nonionic. In the present work, the multistage foam fractionator with bubble caps was operated in continuous steady-state mode. The removal of three types of surfactants from water at feed concentrations at or below the CMCs was studied. The effects of air flow rate, foam height, surfactant feed concentration, and the number of stages on separation efficiency were determined. Auxiliary properties such as foam wetness, foamability (foam formation), and foam stability were also measured to aid in the interpretation of the column results (21–22). These properties, therefore, can be used to interpret the efficiency of separation of different surfactants (14).

## EXPERIMENTAL

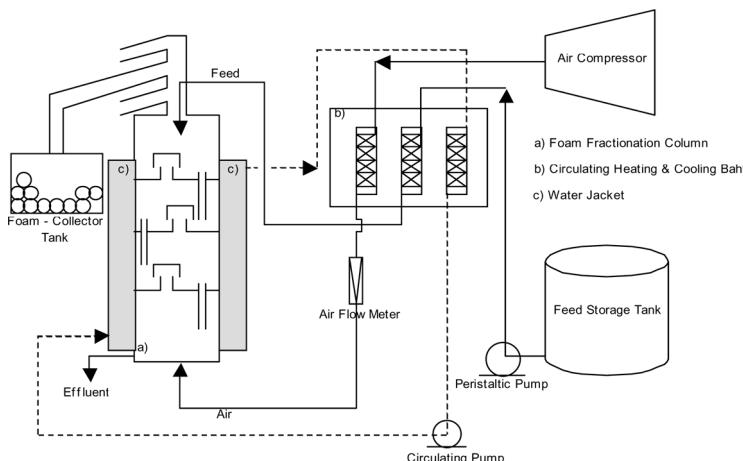
### Materials

Cetyl pyridinium chloride (n-hexadecylpyridinium chloride or CPC) with more than 99% purity, a cationic surfactant, was obtained from Zealand

Chemical. Sodium dodecyl sulfate (SDS), an anionic surfactant with 96.28% purity, was purchased from Kao Industry. Polyoxyethylene (20) sorbitan monolaurate (Span80), with more than 97% purity, a nonionic surfactant known as Rheodol, was purchased from Kao (southeast Asia) Pte Ltd. All chemicals were used as received. Freshly deionized water was used in all experiments.

## Methods

Figure 1 illustrates the experimental set-up of the multistage foam fractionation unit used in this study. The multistage foam fractionation column was comprised of a water-jacketed stainless-steel cylinder having a jacket diameter of 30 cm, an internal column diameter of 20 cm, and a tray spacing of 15 cm. Each tray had 16 bubble caps with a weir height of 5 cm and a cap diameter of 2.5 cm. A sample port was located at the base of each tray for taking liquid samples. Each tray had a glass window for visual observation. Three foam heights of 30, 60, and 90 cm from the top tray of the column were studied. The dimensions and configuration of the multistage foam fractionation column was shown in our previous work (4). A feed solution containing each surfactant at a fixed concentration was continuously fed into the multistage foam fractionation column by using a peristaltic pump at different flow rates in a range of 20–200 mL/min ( $0.7215\text{--}5.77\text{ L/min m}^2$ ) and the feed solution entered the column at the top position of the highest tray. The pressurized air flow



**Figure 1.** Schematic of experimental multistage foam fractionation system.

rate was regulated by a rotameter in the range of 30–90 L/min and was introduced to the bottom of the column. The column operating temperature was held constant at 25°C by using a circulating cooling-heating bath to circulate water through the water jacket around the column. The studied system was first operated to determine the time to establish steady state, which was around 20 hours. Steady state was ensured when all measured parameters were invariant with time. The result shows that the multistage foam fractionator requires a much longer time to reach steady state as compared to only 6 h reported in the previous study for the single-stage unit (6). After the studied system was operated longer than the steady state time of 20 hours, the foamate at the top of the solution was collected at three different heights (30, 60, and 90 cm) from the top tray of the column. In each stage or tray, the rising foam produced was forced to pass through the bubble caps of the upper tray. As a result, the foam was burst and mixed with the liquid in the upper tray, resulting in the surfactant transfer in the upward direction. Except for the highest tray, the produced foam was allowed to flow out from the column at different foam heights. Hence, the foam in this multistage foam fractionator can be analogous to the vapor phase in a fractionated distillation unit. The foam collected was frozen, thawed, and then weighed to measure the mass and volume of the collapsed foamate at room temperature (25–27°C), which are used to indicate the foam wetness (g of collapsed foam solution/L of foam) and foam production rate (mL/min). The samples of the feed solution, the collapsed foamate, and the effluent were analyzed for surfactant concentration. The concentration of SDS was measured by a total organic carbon analyzer (TOC) (Shimadzu, TOC-5000A). The concentration of CPC was measured by using a UV-Visible spectrophotometer (Perkin Elmer, Lambda 10) at a wavelength of 260 nm. The concentration of Span80 was measured by the TOC. The fractionation column was thoroughly cleaned with distilled water before starting the next experiment. All of the experiments were performed at least three times to ensure reproducibility of the results and the mean values of the experimental data were taken to evaluate the process performance in the recovery of these three surfactants. By performing mass balance of the surfactant, the average error was in the range of 5–13%.

In addition, the critical micelle concentration (CMC) of each surfactant was determined from the concentration where the surface tension versus surfactant concentration shows an abrupt change in the slope. The measurement of surface tension values of solutions containing different surfactant types and concentrations was carried out by using a Du-Nouy ring tensiometer (Kruss, K10 T).

Moreover, experiments to measure foamability and foam stability were conducted by using a glass column having an internal diameter of

5 cm and a height of 100 cm. A quantity of 250 mL of solution containing different surfactant types and a fixed surfactant concentration of 50% of its CMC was poured into the column and then the solution was sparged with a constant air flow rate of 0.35 L/min. The foam height was measured as a function of time until the maximum foam height was reached at 90 cm. The time required to obtain the maximum foam height of 90 cm is used to express the foamability of the system, which indicates the ability to generate foam. To quantify foam stability, the air introduced into the column was terminated, and the foam height versus time was then measured. The foam stability is expressed in terms of time required for the complete collapse of foam. All experiments were carried out at room temperature (25–27°C).

## Calculations

The enrichment ratio of surfactant is defined as the concentration of the surfactant in the collapsed foam (foamate) divided by the surfactant concentration in the influent solution as given by:

$$\text{Enrichment ratio} = S_f/S_i,$$

where  $S_f$  and  $S_i$  are the surfactant concentrations in the foamate and in the influent, respectively.

Separation efficiency of the surfactant is defined by the removal fraction, as in the following equation:

$$\text{Removal fraction} = (Q_i S_i - Q_e S_e) / Q_i S_i,$$

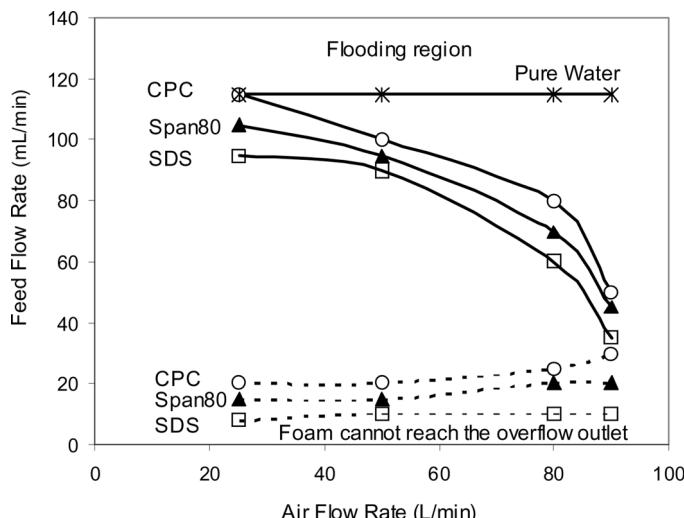
where  $S_e$  is the surfactant concentration in the effluent and  $Q_i$  and  $Q_e$  are the flow rates of the influent and the effluent, respectively.

Both a high enrichment ratio and high separation efficiency are desirable for maximizing surfactant recovery, which implies that a foam fractionator should be operated to achieve a very high enrichment ratio of surfactant with a very low flowrate of foamate produced, as well as a very low surfactant concentration in the effluent.

## RESULTS AND DISCUSSION

### Operational Zones

To operate a multi-stage foam fractionator successfully, one has to consider two important process constraints: foamability (foam formation) and flooding. To achieve surfactant separation, a sufficient air flow rate

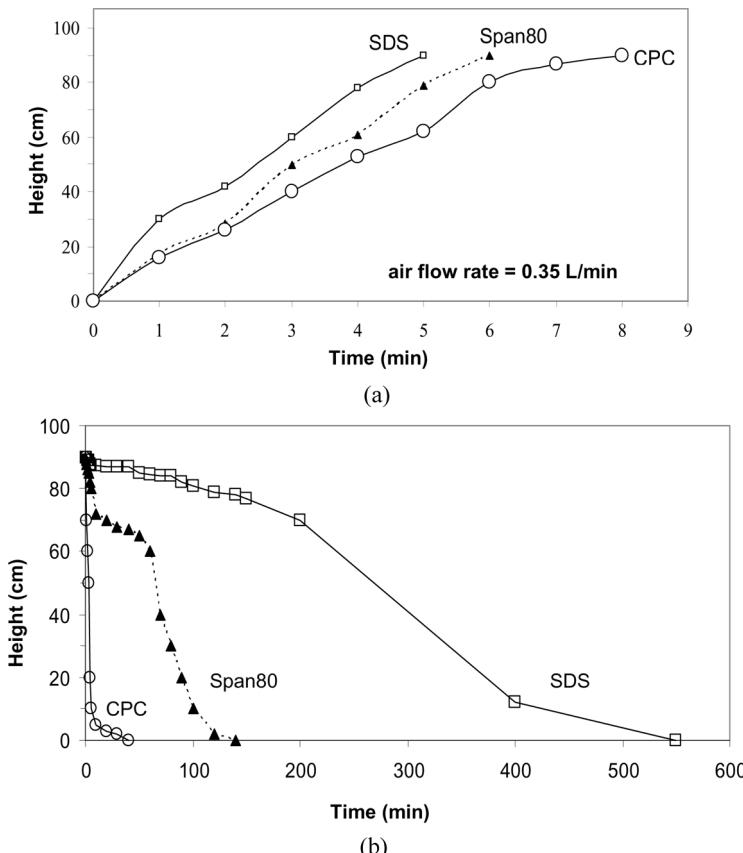


**Figure 2.** Flooding points and operating zones of the multi-stage foam fractionation column for the three surfactants operated at a surfactant concentration = 50% of CMC, foam height = 60 cm, and number of stages = 3.

is needed to produce foam to reach the foam outlet of the top stage. Meanwhile, the flooding of the solution in the column may interrupt or reduce the separation efficiency if the system is operated under a very high flow rate of air and/or a very high feed flow rate. Figure 2 shows the operational regions with the two boundaries of the no-foam regions and the flooding regions for three different types of surfactants. As shown in Fig. 2, for any given surfactant type, the liquid flooding in a stage depends on both the liquid flow rate and the air flow rate. Interestingly, as compared to the pure water system, in the presence of any surfactant, the flooding boundary clearly appeared at a lower feed flow rate or air flow rate since the produced foam can retard the liquid downflow in the system. Interestingly, for all studied surfactants, the foam formation was found to be governed mainly by the feed flow rate. This can be explained in that a quantity of foam produced reaches a maximum value at a high air flow rate because the studied system contained a very low surfactant concentration (50% of CMC), leading to the depletion of surfactant in the system. The operational zone of each surfactant is quite similar, but both the boundary lines for flooding and foam forming are slightly different among these three surfactants, indicating that the presence of any surfactant can affect the operation of a foam fractionator. The differences in the operational zones among the three surfactants will be further discussed in the next section on foam characteristics.

### Foam Characteristics

For successful foam fractionation operation, the system relies on good foam formation ability as well as high foam stability. Figure 3 shows both the foamability and the foam stability of the three studied surfactants. The critical micelle concentrations (CMC) of the three surfactants were determined from the concentration where the surface tension versus surfactant concentration shows an abrupt change in slope. The CMC values of anionic surfactant (SDS), cationic surfactant (CPC), and nonionic surfactant (Span80), are  $8.2\text{ mM}$ ,  $0.9\text{ mM}$ , and  $8.41 \times 10^{-3}\text{ mM}$  ( $2.306\text{ g/L}$ ,  $0.322\text{ g/L}$ , and  $5.405 \times 10^{-3}\text{ g/L}$ ), respectively. Molecular



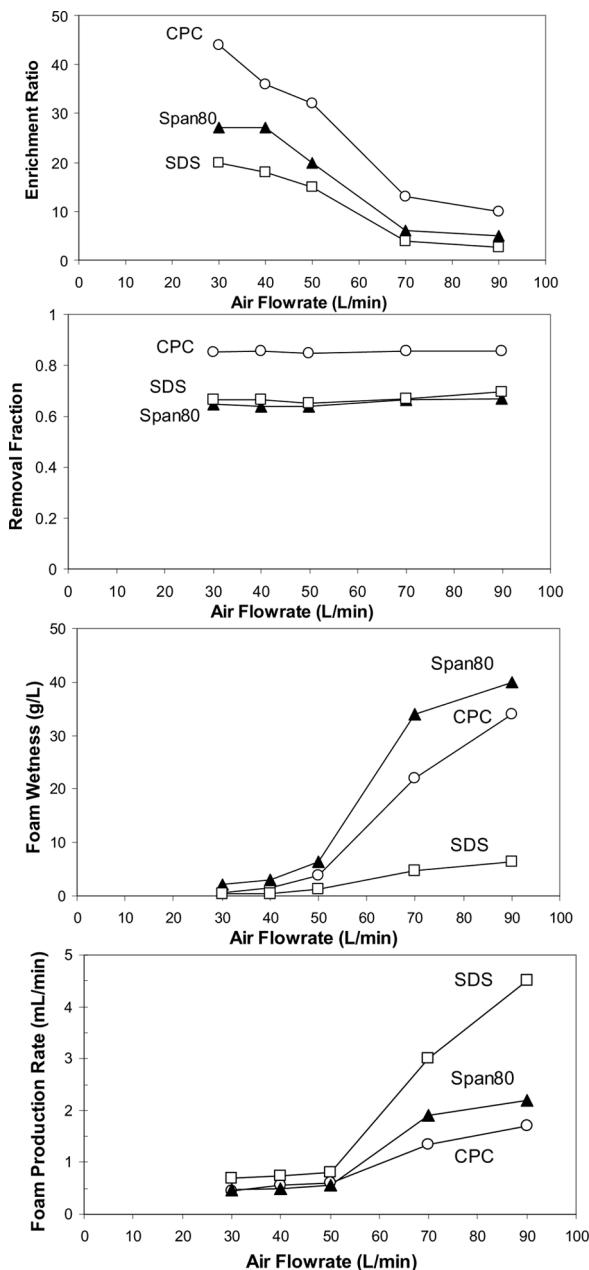
**Figure 3.** Foamability and foam stability of the three surfactants (initial surfactant concentration = 50% of CMC).

weights of SDS, CPC, and Span80 are reportedly 288.38, 358.01, and 643, respectively. The SDS system was found to have both the highest foamability and foam stability among the three studied surfactants, while the CPC system gave the lowest ones.

The operational zone of each surfactant, as shown in Fig. 2, was used to operate the multistage foam fractionator in this study. In order to determine the effects of all process parameters, the system was initially operated under base conditions (air flow rate = 50 L/min; feed flow rate = 20 mL/min; foam height = 60 cm; surfactant feed concentration = 50% of CMC; temperature = 25–27°C and number of trays = 3) and only one process parameter deviated from the base conditions at a time.

### Effect of Air Flow Rate

To observe the effect of air flow rate, a feed flow rate was fixed at a constant of 20 mL/min, which is located in the operational zone. The effect of air flow rate on the surfactant separation performance of the three surfactants is shown comparatively in Fig. 4. For any given surfactant system, an air flow rate lower than the minimum limit (30 mL/min) caused a low production of foam with low foam stability, which collapsed before reaching the overhead outlet at the top of the column. In contrast, an air flow rate greater than the maximum limit (90 mL/min) could not be used because of the flooding effect. From Fig. 4, for any given surfactant type, the enrichment ratio of the surfactant decreases drastically, but the surfactant removal fraction slightly increases with increasing air flow rate. The results can be explained in that an increase in the air flow rate results in more air bubbles being available to generate foam, leading to an increase in the foam production rate confirmed experimentally (see Fig. 4) and this can be explained in that the surface area of the air-water interface simply increases with the increasing foam production rate, leading to more surfactant molecules per unit volume being adsorbed at the air/water interface of the foam. Meanwhile, an increase in the air flow rate results in a higher volumetric rate of foam and a wetter foam, as also confirmed experimentally (see Fig. 4), which, in turn, leads to a lower surfactant enrichment ratio. The results can be explained in that an increase in the air flow rate directly increases the foam production rate, resulting in the increase in the water fraction in produced foam. Consequently, the foam produced becomes wetter or contains more water when the air flow rate increases. Changes in foam bubble size may also cause observed effects, but could not be measured here. The observed effect of the air flow rate is in good agreement with literature (8,12). In a comparison among the three types of the studied surfactants, CPC has the highest



**Figure 4.** Effect of air flow rate on separation efficiency of the three surfactants (feed flow rate = 20 mL/min; foam height = 60 cm; surfactant feed concentration = 50% of CMC; and, number of trays = 3).

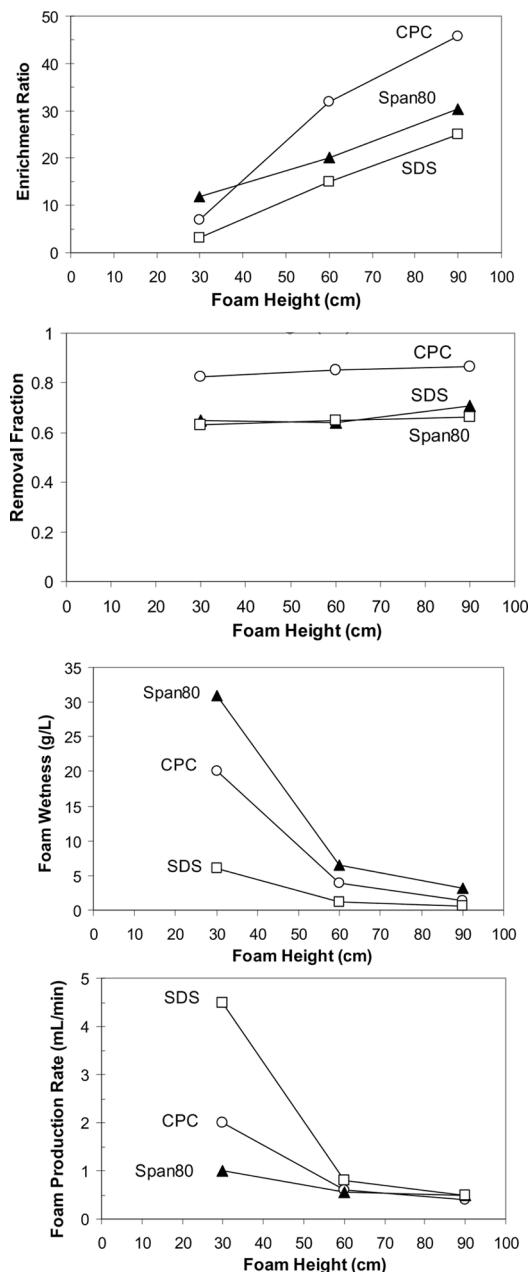
separation efficiency while SDS has the lowest in terms of both removal fraction and enrichment ratio. This can be explained from the results of the foam characteristics and the foam production rate, as shown in Fig. 3. For all three studied surfactants, both the foam production rate and the foamability were almost the same but the foam stability of CPC was the lowest, and that of SDS was the highest. The foam stability of the SDS is higher than that of the CPC and the enrichment ratio of the CPC is higher than that of the SDS, as shown in Fig. 3. Since the feed concentration of the SDS was the highest, it caused the lowest SDS removal fraction. In the case of Span80, its enrichment ratio and removal fraction lie between those of CPC and SDS, which corresponds to the foam characteristic results.

### Effect of Foam Height

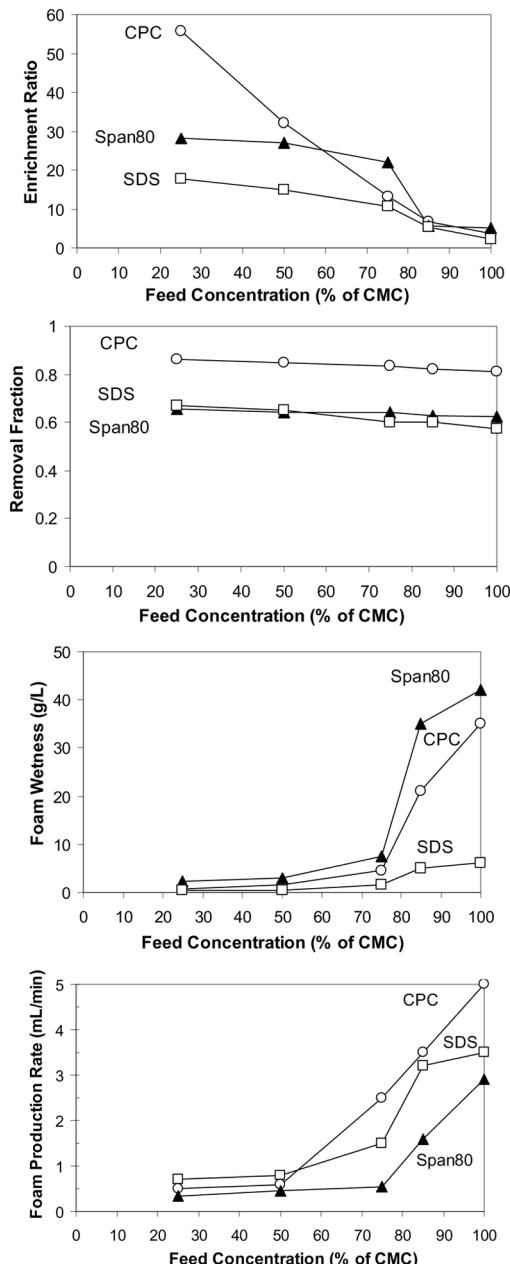
Figure 5 shows the effects of foam height on the separation performance of the three surfactants. For any given surfactant type, an increase in foam height resulted in increasing the enrichment ratio and led to a subtle decrease in the removal fraction. This is because an increase in foam height leads to a longer foam residence time, which allows more drainage of the liquid in the films, as confirmed by the sharp decreases in both foam wetness and foam production rate (see Fig. 5). Hence, a dryer foam with a greater enrichment ratio is obtained. Meanwhile, the removal fraction decreased slightly with increasing foam height because of the increased rate of foam collapse due to foam drainage (decreased foam production rate). Figure 5 also shows comparatively the effect of foam height on surfactant recovery among the three different surfactants. For any given foam height, CPC had the highest of both enrichment ratio and removal fraction followed by Span80 and SDS. The same explanation, as mentioned before for the effect of air flow rate on the separation of different surfactant types, can be used for explaining the effect of foam height.

### Effect of Feed Concentration

Figure 6 illustrates the effects of feed concentration of the surfactant on the surfactant separation performance, as well as the foam production rate and foam wetness of the three types of studied surfactants. For any given surfactant type, the enrichment ratio of the surfactant decreased remarkably with an increase in the feed concentration of the surfactant. At surfactant concentrations much lower than its



**Figure 5.** Effect of foam height on separation efficiency of the three surfactants (air flow rate = 50 L/min; feed flow rate = 20 mL/min; surfactant feed concentration = 50% of CMC; and, number of trays = 3).



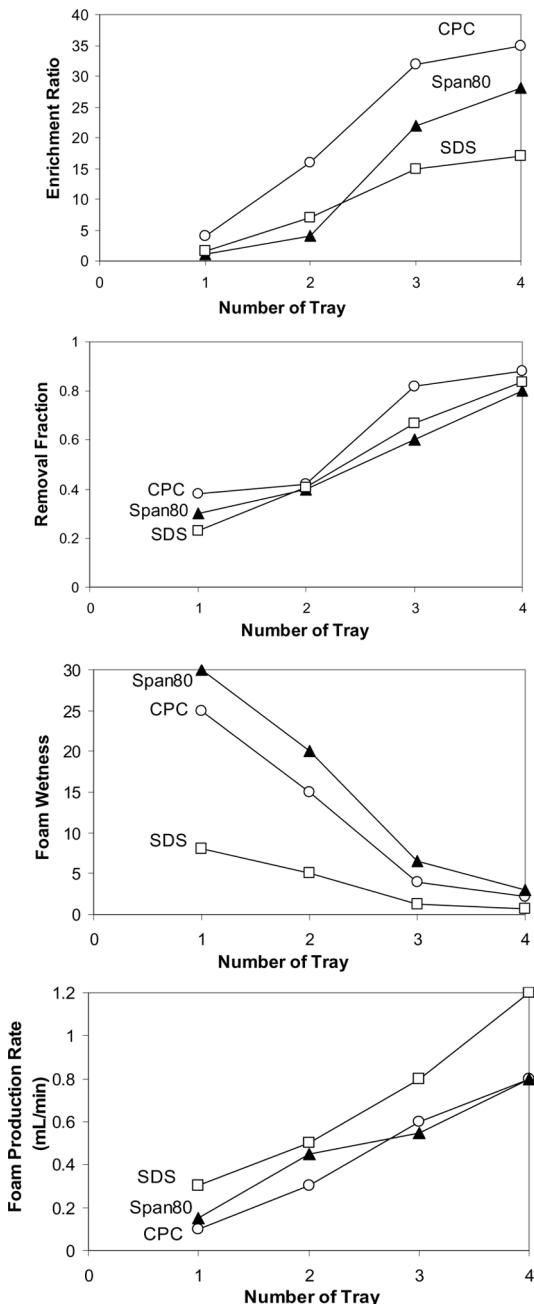
**Figure 6.** Effect of influent surfactant concentration on separation efficiency of the three surfactants (air flow rate = 50 L/min; feed flow rate = 20 mL/min; foam height = 60 cm; and, number of trays = 3).

CMC, an increase in surfactant concentration results in an increase in the excess surface concentration of the surfactant, leading to a decrease in the surface tension. The former effect tends to increase the enrichment ratio, whereas the latter effect results in lower rates of drainage and higher rates of foam production and foam wetness, thus leading to lower enrichment ratios as seen in Fig. 6. The former effect seems to be predominant since the enrichment ratio was found to decrease with concentration of surfactant. At high concentrations close to its CMC, both quantities of the generated foam in terms of the foam volume and the collapsed foam volume increase substantially with increasing the surfactant concentration in the feed solution for all the studied surfactants, as shown in Fig. 6. As a result, a highest decrease in the enrichment ratio of the surfactant was found around its CMC. Interestingly, a higher enrichment ratio in multi-stage foam fractionation occurs at lower surfactant concentrations; but this improvement is limited by a minimum surfactant concentration for enough foaming to generate the overhead froth. For any given surfactant type, the removal fraction of the surfactant was almost invariant in the studied range of surfactant concentrations. The result can be explained in that the foam production rate increases proportionally to the feed concentration of the surfactant, known as the Gibb-Marangoni effect, as shown in Fig. 6.

In a comparison among the three different types of surfactants, for any given feed concentration, the cationic surfactant (CPC) gave the highest enrichment ratio among the three surfactants. This was due to the characteristics of the surfactants as explained in the previous section on foam stability. CPC, as a cationic surfactant, gives the lowest foam stability (lower than the anionic and nonionic surfactants) corresponding to the visual observation of larger bubble sizes of CPC than those of the SDS and Span80.

### Effect of the Number of Stages

In order to determine the effect of the stage number, the studied system was operated at a surfactant feed concentration of 50% of CMC, a feed flow rate of 20 mL/min, a foam height of 60 cm, and an air flow rate of 50 L/min, while the stage number was varied from 1 to 4. Figure 7 shows the effect of the number of stages on the surfactant separation efficiency of the CPC, SDS, and Span80. For any given surfactant type, the enrichment ratio and removal fraction of the surfactant increased with increasing number of stages. The results can be explained in that an increase in the number of stages simply increases the foam production



**Figure 7.** Effect of number of stage on separation efficiency of the three surfactants (air flow rate = 50 L/min; feed flow rate = 20 mL/min; foam height = 60 cm; and surfactant feed concentration = 50% of CMC).

rate but decreases the foam wetness, as shown in Fig. 7. This is because an increase in the number of stages simply increases both the residence time of the liquid and the air bubbling rate in the system. In a comparison among these three surfactants, CPC had the highest values of enrichment ratio and removal fraction, while SDS had the lowest. The same explanations for the effect of the feed flow rate can be used to explain the effect of the number of stages.

In a comparison, an increase in the numbers of stages can enhance both the removal and enrichment ratio of the surfactants but the other operational parameters can only increase either the removal or the enrichment ratio. With regard to the results, then the design of a multi-stage foam fractionator should have at least three stages.

## CONCLUSIONS

The use of a multi-stage foam fractionation unit was investigated for the recovery of three types of surfactants: CPC, SDS, and Span80. The surfactant concentration in the feed solution was varied in the range of 50 to 100% of CMC. In a comparison among the operational parameters, air flow rate, feed concentration, and foam height had insignificant effects on the removal efficiency for all the studied surfactants. For any given surfactant type, an increase in foam height increased the enrichment ratio of the surfactant, which was in contrast with the effects of the air flow rate and feed concentration. Interestingly, when the foam fractionation unit had a higher stage number, both the enrichment ratio and the removal of the surfactant increased substantially for all three studied surfactants. Based on these results, multistage foam fractionation can be used for surfactant recovery efficiently at a very low surfactant concentration (lower than its CMC). The effect of stage number was found to be the only operational parameter to actually enhance both enrichment ratio and removal of surfactant. At least three stages are recommended for the design of a multistage foam fractionator.

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